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17 September 2009

Department of the Army
Louisville District, Corps of Engineers
600 Dr. Martin Luther King Jr. Place
Louisville, KY 40202

Attn: Richard Kennard

Subject: Final Remedial Investigation/Feasibility Study (RI/FS) Work Plan for the Open Burn/Open Detonation Area (SVAD-050), Savanna Army Depot Activity, Savanna, Illinois

Dear Mr. Kennard:

ERT, Inc. is pleased to submit this Final RI/FS Work Plan for the Open Burn/Open Detonation Area (SVAD-050), Savanna Army Depot Activity in accordance with the Scope of Work (SOW) from the USACE, dated 7 August 2008. The updated plans incorporate comments from U.S. EPA Region 5 and IEPA, as well as the resolution of these comments made during our conference call on 21 August 2009.

This Final RI/FS Work Plan has been submitted, including two compact disks (CDs) containing the complete RI/FS Work Plan electronic files to the USACE Louisville District, and one hard copy of the document and CD to the installation. Copies of the RI/FS Work Plan, along with CDs, have also been sent to US EPA Region 5 and Illinois EPA, per the request of USACE-Louisville.

The RI/FS Work includes the following appendices, comprising other individual plans related to the work being conducted at SVAD-050:

- Site Safety and Health Plan
- Geophysical Investigation Plan
- Munitions and Explosives of Concern (MEC) Investigation Plan
- Sampling and Analysis Plan
- Quality Assurance Project Plan

The RI/FS Work Plan and QAPP have been reviewed by SAIC, and all plans have been reviewed by the Independent Technical Reviewer.

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We look forward to providing support to USACE in your continuing efforts at SDVA.

Very truly yours,

Michael Dorman
Project Manager

cc: Mr. John Clarke, Savanna Army Depot Activity w/ one hardcopy and one CD
Mr. Joe King, USAEC w/ one hardcopy and one CD
Mr. Tom Barounis, US EPA Region 5 w/ two hardcopies and two CDs
Ms. Charlene Falco, IEPA, w/ two hardcopies and two CDs
Mr. Glenn Earhart, ERT Program Manager w/o enclosures
ERT File

FINAL
WORK PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION AREA (SVAD-050),
SAVANNA ARMY DEPOT
SAVANNA, ILLINOIS

Prepared for

U.S. Army Corps of Engineers
Louisville District

Contract W912QR-08-D-0012
Delivery Order 0001

Prepared by

Earth Resources Technology, Inc.
4890 University Square, Suite 2A
Huntsville, Alabama 35816
(256) 217-8991

September 2009

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REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION AREA (SVAD-050),
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APPROVED BY

9/2/09

Michael Dorman — Date
Project Manager

5/20/09

Glenn Earhart — Date
Program Manager

COMPLETION OF SENIOR TECHNICAL REVIEW

This document has been produced within the framework of the Earth Resources Technology, Inc. (ERT) and ISO Approved 9001-2000 quality management systems certification As such, a senior technical review, as defined in the project Quality Control Plan, has been conducted. This included review of the overall design addressed within the document, proposed or utilized technologies and alternatives and their applications with respect to project objectives, and Department of the Army policy and regulatory guidance and constraints

Raymond D. Bowman 12/19/08

Raymond Bowman, PG Date
Senior Technical Reviewer

COMPLETION OF INDEPENDENT TECHNICAL REVIEW

This document has been produced within the framework of ERT's total quality management system and ISO 9001:2000 certification. As such, an independent technical review, appropriate to the level of risk and complexity inherent in the project as defined in the project Quality Control Plan, has been conducted. This included review of assumptions (methods, procedures, and material used in analyses), alternatives evaluated; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets the project objectives. Comments and concerns resulting from review of the document have been addressed and corrected as necessary.

Wanfang Zhou 5/20/09

Dr. Wanfang Zhou Date
Independent Technical Reviewer

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LIST OF ACRONYMS

°F	Degrees Fahrenheit
ACGIH	American Conference of Governmental Industrial Hygienists
ADR	Automated Data Review
ASR	Archive Search Report
ATF	Alcohol, Tobacco, Firearms, and Explosives
BERA	Baseline Ecological Risk Assessment
BIP	Blow-in-Place
bls	below land surface
BRAC	Base Realignment and Closure
CEHNC	U.S. Army Engineering and Support Center, Huntsville
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
COR	Contracting Officers Representative
DA	Department of Army
dbh	diameter at breast height
DID	Data Item Description
DOD	Department of Defense
DOT	Department of Transportation
DGM	Digital Geophysical Mapping
EDS	Environmental Data Services, Inc.
EOD	Explosive Ordnance Disposal
ERA	Ecological Risk Assessment
ERT	Earth Resources Technology
ESE	Environmental Science and Engineering
EZ	Exclusion Zone
FS	Feasibility Study
ft	feet
GIS	Geographic Information System
GPO	Geophysical Prove Out
HHRA	Human Health Risk Assessment
HTRW	Hazardous, Toxic, and Radioactive Waste
IDNR	Illinois Department of Natural Resources
IDOC	Illinois Department of Conservation
IDW	investigation-derived waste
IEPA	Illinois Environmental Protection Agency
L&D	Lock and Dam
MEC	Munitions and Explosives of Concern
MPPEH	Materials Potentially Presenting an Explosive Hazard
MWH	Montgomery Watson Harza
NCP	National Contingency Plan
NELAP	National Environmental Laboratory Accreditation Program
NHPA	National Historic Preservation Act
NOAA	National Oceanic and Atmospheric Administration
NEPA	National Environmental Policy Act
NRHP	National Registry of Historic Properties

OB	Open Burn
OBG	Old Burning Ground
OD	Open Detonation
PETN	Pentaerythritoltetranitrate
PM	Project Manager
POC	Point of Contact
PP	Proposed Plan
PPE	Personal Protective Equipment
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QCP	Quality Control Plan
RCRA	Resource Conservation and Recovery Act
RDX	Cyclotrimethylenetrinitramine
RI	Remedial Investigation
ROD	Record of Decision
SAIC	Science Application International Corporation
SAP	Sampling and Analysis Plan
SCS	Soil Conservation Service
SEDD	Staged Electronic Data Deliverable
SHPO	State Historic Preservation Office
SOP	Standard Operating Procedure
SOW	Scope of Work
SRA	Soil Retention Area
SSHP	Safety and Health Plan
SUXOS	Senior UXO Supervisor
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
SWMU	Solid Waste Management Unit
TACO	Tiered Approach to Corrective Action Objectives
TCLP	Toxic Characteristic Leaching Procedure
T&E	Threatened and Endangered
TNT	Trinitrotoluene
TRV	Toxicity Reference Value
UMNWR	Upper Mississippi National Wildlife Refuge
USACE	U.S. Army Corps of Engineers
USAEHA	U.S. Army Environmental Hygiene Agency
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
USEPA	U.S. Environmental Protection Agency
USFWS	U.S. Fish and Wildlife Service
UXO	Unexploded Ordnance
UXOQC	UXO Quality Control
UXOSO	UXO Safety Officer
WP	Work Plan
yd^3	cubic yard

1.0 INTRODUCTION

1.1 Project Authorization

Earth Resources Technology, Inc. (ERT) received Contract Number W912QR-08-D-0012 issued by the U.S. Army Corps of Engineers (USACE), Louisville District on 29 September 2008. Task Order 001 was issued for the purpose of performing the work identified in the Scope of Work, which includes a Remedial Investigation (RI) and Feasibility Study (FS) at the Savanna Army Depot Activity (SVDA), Savanna, Illinois, at the Open Burn/Open Detonation (SVAD-050) Site.

1.2 Purpose and Scope

This Work Plan (WP) provides details for Remedial Investigation/Feasibility Study (RI/FS) Activities within Site SVAD-050 at the depot, as described in the Statement of Work (SOW) for Task Order 001 under Contract W912QR-08-D-0012. The objective of the Task Order is to evaluate the nature and extent of potential contamination at SVAD-050 from past practices, determine if the potential contamination poses threat to human and ecological receptors, and develop a feasibility study to evaluate and select an alternative to minimize any potential threats. As part of the characterization phase of this study, the areas to be investigated will be cleared of vegetation, the ground surface will be cleared of any Munitions and Explosives of Concern (MEC); a geophysical survey will be conducted; test pits will be dug at select anomalies; and samples will be collected and analyzed from surface and subsurface soil, groundwater, surface water, and sediments. The results of the investigation will be documented in a RI report, along with a FS, Proposed Plan, and a Record of Decision (ROD). These documents will occur in separate phases of the project. A 45-day review period has been planned for USEPA and IEPA on each of the documents.

1.3 Regulatory Status

Work completed under this task order will be consistent with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and the National Contingency Plan (NCP), Executive Order 12580. In addition, all activities involving work in areas potentially containing MEC hazards shall be performed in consistent with U.S. Army Engineering and Support Center, Huntsville (CEHNC), Department of the Army (DA), and Department of Defense (DOD) requirements regarding personnel, equipment and procedures. The work required under this task order falls under the Base Realignment and Closure (BRAC) Program. In 1995, SVDA was placed on the BRAC list. A Federal Facilities Agreement for SVDA exists with the U.S. Environmental Protection Agency (USEPA) and the State of Illinois Environmental Protection Agency (IEPA).

Public law requires that government facilities subject to closure and subsequent reuse be subject to cleanup under CERCLA. Because MEC is a safety hazard and may constitute an imminent and substantial endangerment to the site personnel and local populace, the provisions of 29 Code of Federal Regulations (CFR) 1910.120 shall apply to all actions taken at this site.

1.4 Work Plan Organization

The RI/FS work plan is organized as follows:

- Section 1 provides a summary of the project, describes the physical/environmental setting, and presents an overview of historical investigations at the site.
- Section 2 presents the project organization, including key ERT personnel, subcontractor roles and responsibilities and key subcontractor staff, as well as general site access and control
- Section 3 provides an overview of project reporting and deliverables
- Section 4 presents the details of the Risk Assessment methodology
- Section 5 presents details of the FS, Proposed Plan, and ROD process.
- Section 6 presents the Environmental Protection Plan
- Section 7 presents references
- Appendix A provides the Site-Specific Safety and Health Plan for the RI/FS field activities
- Appendix B presents the Geophysical Investigation Plan
- Appendix C presents the MEC Investigation Plan
- Appendix D provides the Sampling and Analysis Plan (SAP). The Field Sampling plan contained within the SAP should be reviewed in conjunction with Appendix E.
- Appendix E provides the Quality Assurance Project Plan (QAPP).
- Appendix F provides resumes of additional key technical staff that were not included in the Quality Control Plan.

1.5 Site Location

Prior to base closure, SVDA was a 13,062-acre installation located approximately 7 miles north of Savanna, Illinois, in Jo Daviess and Carroll Counties, Illinois. The installation is surrounded primarily by agricultural land, with the communities of Hanover, Illinois to the east; Bellevue, Iowa to the northwest; and Savanna, Illinois to the southeast. Figure 1-1 provides the general location of SVDA.

1.6 Site History

1.6.1 Open Burn/Open Detonation Area (SVAD-050)

The area has been used since approximately 1936 for the demolition of various explosives, such as trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), and pentaerythritoltetranitrate (PETN), by detonation. SVAD-050 covers approximately 7 acres. Several distinct areas within SVAD-050 have been identified based on historic use and the investigation approach outlined in this Work Plan. These areas are the Open Detonation (OD) Area, the Open Burn (OB) Area, and the roads interconnecting the two sites. These areas are identified on Figure 1-2.

1.6.1.1 Open Detonation Area

The OD grounds were used to detonate obsolete projectiles and ordnance. OD has taken place using two demolition configurations. Early demolitions beginning in 1946 took place on six

demolition pads all located within the current boundary of the OD Area. Later, the site was re-configured so that all demolition took place in earthen pits constructed of uncompacted fill material (loose soil). The OD Area has two approximately 300-foot long berms between which the pits were located. There were three pits per berm and each pit was at least 4 feet (ft) deep and was covered with at least 4 ft of earth. As with OB operations, all wastes treated by open detonation were characteristic wastes only, exhibiting the characteristic of reactivity and/or toxicity, as defined by the Toxic Characteristic Leaching Procedure (TCLP) (NUS, 1993). After each series of shots (up to six pits), the surrounding area was searched for unexploded material. Materials such as lumps of explosives or unfuzed ammunition were collected and prepared for the next round of shots. Fuzed or unstable ammunition, as determined by the supervisor and/or quality assurance (QA) representative, was detonated in place.

Prior to BRAC, the amount of activity at the open detonation area was determined by the amount of funding available for demolition of unserviceable ordnance. The OD area was used most recently for demolition of unexploded ordnance (UXO) recovered from MEC derived from restoration activities. The Resource Conservation and Recovery Act (RCRA) interim status permit allowed OD demilitarization operations until October 2008. The OD area is covered by tall grasses and saplings, and is regularly flooded by the Mississippi River in the spring.

1.6.1.2 Open Burn Area

The OB pad was used to burn obsolete or unstable propellants. Standard procedures required that propellants be placed in pans prior to burning. Each pan was situated on a concrete pad and elevated above the pad by steel I beams. After the propellants were burned, ashes were collected from the pans and placed into a Department of Transportation (DOT) approved drum, which then was transferred to Building E704 where it was stored between OB activities. All wastes treated by OB were characteristic wastes only, exhibiting the characteristics of reactivity and/or toxicity as defined by the TCLP (NUS, 1993). No listed wastes were treated at the OB pads. If the residuals were non-hazardous, they were disposed of as solid waste. If the TCLP tests showed that the residuals were characteristic for toxicity, they were disposed of offsite at a permitted RCRA disposal facility. The OB pads were not used except for emergencies. The last burn occurred in 1985. This area is currently covered by tall grasses and is otherwise open.

1.6.1.3 Other Areas

Also within the boundaries of SVAD-050 are approximately 900 linear ft of gravel road that have been used to transport munitions and explosives to and from disposal/demolition areas.

1.6.2 Old Burning Ground (SVAD-009-R-01)

The Old Burning Ground (OBG) is located in the west-central portion of SVDA, approximately 2,100 ft from the main channel of the Mississippi River (see Figure 1-3). The OBG encompasses a total area of approximately 120 acres and includes a former burn area for explosives (Site 13) and a burn site (Site 14). Site 13 includes multiple burn pads and cages scattered within the OBG that were used approximately between 1930 and 1985 (Science Applications International Corporation [SAIC], 2004). Site 14 consists of a single burn site that was used between 1942 and 1945 to burn 12 - 155 mm H-filled projectiles (SAIC, 2004). SAIC is currently performing RI/FS activities at the OBG.

1.7 Environmental Setting

1.7.1 Climate

SVDA is located in an area with a typical continental climate, characterized by cold winters; warm summers; and frequent short-term fluctuations in temperature, humidity, cloudiness, and wind direction. The winds are controlled primarily by storm systems and weather fronts that move eastward and northeastward across the area.

The National Oceanic and Atmospheric Administration (NOAA) has prepared a climate summary for Bellevue, Iowa (0.5 miles west of SVDA), for the years between 1948 and 2002 (NOAA, 2002). Average monthly temperatures range from 10.4 degrees Fahrenheit (°F) to 84.2°F and drop below 0°F an average of 15 times per year (NOAA, 2002). The average monthly temperatures are at or near freezing between December and February. The soil freezes to a depth of approximately 2 ft below land surface (BLS) and may remain snow-covered for weeks at a time. In contrast, the area experiences a temperature of at least 90°F an average of 11 days each year during the summer months (NOAA, 2002). Extreme heat and humidity rarely last more than a few days because of cool air masses moving southward from Canada (Dames & Moore, 1994).

The mean total annual precipitation in Bellevue, Iowa between 1949 and 2001 was 33.7 inches (NOAA, 2002) with a maximum of 45 inches (1951) and a minimum of 20.4 inches (1988). The driest months range from December to February with average rainfall from 1.2 to 1.6 inches per month and the wet season occurs from May to September with average monthly precipitation from 3.6 to 4.4 inches per month (NOAA, 2002). The summer months are characterized by short duration, localized showers; however, summer thunderstorms may be severe and are sometimes accompanied by hail or destructive wind. Flooding frequently occurs with the breakup of river ice in the spring, especially if a thick snow cover has been removed by rain or unseasonably warm temperatures (Dames & Moore, 1994).

SVDA has experienced measurable snowfall 3 to 25 times per year between 1948 and 2002, with an average of 16 snowfalls per year. Average annual snowfall in Bellevue, Iowa from 1949 through 2002 was 28.4 inches (NOAA, 2002) ranging from 6.8 to 61.5 inches. The highest snowfall totals occur between December and March with lesser amounts in November and April. Heavy snows of 10 to 14 inches occur once or twice a year, and prevailing winds tend to pile the snow into high drifts. Moderate to heavy ice storms occur annually. Damaging winds may develop into tornadoes at any time of year, but are more likely to occur from March through June (Dames & Moore, 1994).

1.7.2 Topography

The SVAD-050 consists of land slightly elevated above the surrounding Mississippi River backwater areas. However, because of past SVAD-050 site activities, the area contains a number of man-made features, the most notable of which are the elevated burn pads and roadways. The elevation of these features averages 596 ft above mean sea level (msl). Mississippi River gauge data from Lock and Dam (L&D) No. 12 indicate that seasonal floodwaters have exceeded the average elevation (596 ft above msl) of the raised burn pads and roadways 19 times since 1950, submerging most of the SVAD-050 during these flood events. Since 1997, one major flood

event occurred in April 2001, with an elevation of 602.78 ft above msl, completely submerged the SVAD-050 (SAIC, 2004).

In contrast to the elevated burn pads and roadways, the adjacent land areas are generally low lying, with an average elevation of 592 ft above msl. These areas include the interior of the oval-shaped road and the marginal north, northeast, and west-central portions of the SVAD-050.

The eastern edge of the SVAD-050 is bordered by Straight Slough, which connects large areas of the backwater system to the Mississippi River and receives flow from Crooked Slough, approximately 2.3 miles upstream of the SVAD-050. The western edge of the SVAD-050 is bordered by a minor slough, which branches off from Crooked Slough approximately 3,400 ft northwest of the northern SVAD-050 limit. During rising river stages, surface water flow in Straight Slough reverses direction to the north. This reversed flow occurs for a short period until the water levels of the Mississippi River and the backwater system reach equilibrium.

1.7.3 Soils

The U.S. Soil Conservation Service (SCS) classifies the natural surface soil around the SVAD-050 as hydric soils in the Birds Series, consisting of dark brown to dark gray organic, soft, silty clay. However, most of the surface soil at the SVAD-050 has been graded and reworked during past site-related activities, and much sandy fill has been added. The surface soil predominantly consists of fill that is brown, fine- to medium-grained, moist, silty sand commonly mixed with some ordnance-related debris or burn debris. The road surface and some of the burn pads generally contain 6 inches of compacted aggregate (Dames & Moore, 1998).

1.7.4 Stratigraphy

Geologic literature (Willman and Frye, 1970) suggests that the region encompassing the SVAD-050 contains recent alluvial deposits of the Cahokia Formation (silty sand, silt, and clay) at the surface. The Cahokia Formation is underlain by the cleaner and coarser sands of the Parkland Formation, which is underlain by the relatively thin (generally less than 15-foot thick) coarse sand and gravel of the Henry Formation. The Henry Formation is reported to directly overlie dolomite bedrock (Galena Formation).

The undisturbed stratum underlying the layer of waste debris and fill is composed of alluvial deposits, possibly of the Cahokia Formation. These deposits consist of dark brown, moist to wet, silty sand (60 to 80 percent fine and medium, 20 to 40 percent silt and clay). Results of physical soil tests from well borings indicate that the amount of silt decreases with increasing depth. A layer of pale reddish brown stiff clay occurs locally in the extreme southwest part of the SVAD-050. Approximately 5 ft of yellowish brown moderately well-sorted sand with a trace of silt—variably consisting of silt, sand, and clay—underlies the shallow subsurface. Approximately 10 percent silt grades back into the sand at 15 ft BLS in the eastern area of the SVAD-050, while silt occurs only in trace amounts greater than 15 ft BLS in the western area.

1.7.5 Geology

The Dames & Moore OBG RI (Dames & Moore, 1998) presented groundwater data compiled during the Dames & Moore investigations conducted in 1992 and 1993. Geologic conditions at the SVAD-050 are detailed from soil borings drilled by Dames & Moore in 1992 and 1993. The

hydrogeologic information has been updated to include both the Dames & Moore groundwater investigations and the SAIC groundwater investigations conducted from 2000 through 2001.

Geologic and hydrogeologic conditions underlying the OBG area have been characterized during the Dames & Moore investigations at the site (Dames & Moore, 1994) and through supplemental monitoring conducted by SAIC between March 2000 and March 2003. The shallow aquifer underlying the site area consists of fine- to coarse-grained sand with interlayered clay encountered predominantly in borings associated with Site 14.

The hydrogeologic properties of the aquifer underlying the SVAD-050 area have been evaluated through manual and continuous groundwater elevation monitoring conducted during the Dames & Moore and SAIC investigations and through site-specific slug testing conducted during the Dames & Moore investigation (Dames & Moore, 1994). Manual groundwater monitoring was conducted in the Site 13 wells between April and August 1992 and in all OBG wells between March 2000 and March 2003. The observed range in groundwater elevation was from 585.3 to 593.0 ft above msl, averaging 587.8 ft above msl. The depth to groundwater ranged from 0 to 12.8 ft BLS. The Mississippi River stage at L&D No. 12, located approximately 3 miles upstream from the OBG, ranged in elevation from 583.2 to 602.74 ft above msl between March 2000 and March 2003 with an average river elevation of 587.8 ft above msl (USACE, 2003) during this time period.

1.7.6 Natural Resources

1.7.6.1 Endangered / Threatened Species

SVDA has documented two federally listed species and 36 state-listed endangered and/or threatened species, including 14 plant species and 22 animal species. So as to minimize potential impacts to threatened and endangered (T&E) species to the extent practicable resulting from necessary site activities, the appended Savanna Army Depot - Endangered & Threatened Species – Field Identification Manual, April 2004 has been prepared. This manual is intended to afford field personnel an available reference source to avoid T&E species, if possible, during the completion of necessary activities.

The U.S. Fish and Wildlife Service (USFWS), Rock Island Field Office, and the Illinois Department of Natural Resources (IDNR)—previously the Illinois Department of Conservation (IDOC)—were consulted for information on T&E faunal species that potentially occur at or near SVDA. This survey resulted in the identification of several T&E bird, mammal, reptile, amphibian, and fish species that are potentially present at or near the SVDA.

Of the Federal and state T&E species, the great egret (*Casmerodius albus*), brown creeper (*Certhia americana*), and river otter (*Lutra canadensis*) have the greatest potential to occur in the vicinity of the SVAD-050. The state-listed great egret is known to nest within the vicinity of the SVAD-050 and has occasionally been observed in an abandoned great blue heron rookery located at Prairie Lake, north of the SVAD-050. Great egrets were observed in Straight Slough in the spring of 1997 by a Montgomery Watson Harza (MWH) environmental biologist (Montgomery Watson, 1997). The state-listed threatened brown creeper has been documented as a nesting species at SVDA (IDOC, 1991), although it was not observed during the 1992 or 1997 field investigations. Finally, the river otter has been observed in the backwaters of Mississippi

River Pool 13 and in the slough system adjacent to the northern region of SVDA. Although sufficient habitat is present for the river otter at the SVAD-050, no known documentation exists of otter activity in the adjacent aquatic system apart from the potential sighting during the 1997 survey (Montgomery Watson, 1997).

According to IEPA's records, SVAD-050 is also in the vicinity of the following State-listed species (IDNR, 2008, 2009): Butterfly mussel-*Ellipsaria lineolata* (threatened), Black Sandshell mussel-*Ligumia recta* (threatened), and Upland Sandpiper-*Bartramia longicauda* (endangered)

1.7.6.2 Sensitive Plant Communities

Six major terrestrial areas were delineated during a survey conducted at the SVAD-050 in the summer of 2000 (MWH, 2003). These areas included: (1) developed/heavily disturbed; (2) grass /forb mix; (3) willow complex; (4) floodplain forest; (5) low floodplain meadow; and (6) semi-upland forest, the latter being very limited. Dames & Moore characterized the open water vegetative community in their 1992 field investigation. Each of the six major terrestrial vegetative areas and the open water vegetative community are described below.

- The developed/heavily disturbed area consists of buildings, roads, and other areas basically devoid of vegetation or with only a marginal plant component. Black medic (*Medicago lupulina*), common ragweed (*Ambrosia artemisiifolis*), and muletail (*Erigeron spp*.) are the most common plant species in the few areas having any vegetative cover, but this probably should not be considered a true habitat type. Developed/paved areas comprise approximately 5 percent of the land at the SVAD-050 (MWH, 2003).

- The grass/forb mix area is a large area, divisible into two components. The first, and most limited, is the more recently disturbed area, generally located alongside roads, on recently abandoned roads, or recently mowed or graded areas. It tends to occur on slightly higher elevations and is the drier portion of this habitat. No species or group of species could be called dominant, although some are quite common in localized areas. These include common ragweed (*Ambrosia artenisiifolia*), muletails (*Erigeron spp*.), partridge pea (*Cassia fasciculate*), thistle (*Cirsium arvense, C. vulgare, and Sonchus oleraceua*), switch grass (*Panicum virgatum*), tall white clover (*Mililotus alba*), daisy fleabane (*Erigeron annuus*), goldenrods (*Solidago spp*.), western ragweed (*Ambrosia coronopifolia*), and horsemint (*Monarda punctata*). Many other species occur but are not dominant, even over small areas. The roadside grass/forb mix comprises approximately 5 percent of the land area at the SVAD-050.

  The second, and by far most common, grass/forb component is generally located between the roadside grass/forb areas and the floodplain forests. In some places, it includes scattered trees (mostly honey locust [*Gleditsia tricanthos*]), but in many areas is open. It appears to have not been heavily disturbed in the last couple of years, and is generally at a little lower elevation and, therefore more moist than the roadside habitat. The moist grass/forb mix comprises approximately 14 percent of the land area at the SVAD-050. This area is also extremely diverse, with no single species qualifying as dominant, although late goldenrod (*Solidago gigantea*) commonly occurs in most areas. A number of species are "dominant" in small patches in this habitat type. In slightly drier areas,

these include goldenrods (*Solidago spp., especially S. gigantea*), grape (*Vitis sp.),* sage (*Artemesia leudoviciana*), verbena (*verbena stricta*), St. John's wort (*Hypericum sp*.), mixed grasses (especially bluegrass [*Poa pratensis*], dropseed [*Sporobolus sp.*], and switchgrass [*Panicum virgatum*]). In slightly lower, moister areas, rice cutgrass (*Leersia oryzoides*), germander or wood sage (*Teucrium canadense*), and smartweed (mostly *Polygonum pennsylvanicum*) are common. In some locations in the grass/forb mix, willows (mostly sandbar willow [*Salix interior*]) are beginning to invade.

- Dense willow forest is common on much of the SVAD-050. This varies from approximately 15- to 20-foot tall stands down to areas just being taken over by dense seedlings. The dominant tree is the sandbar willow (*Salix interior*), although scattered black willow (*Salix nigra*) and occasional silver maple (*Acer saccharinum*) were noted. The willow complex comprises approximately 16 percent of the land area at the SVAD-050. The understory depends on the development stage and elevation of the willow forest. In the tallest, developed willows, smartweed (*Polygonum spp*.), nettle (*Urtica dioica*), and cutgrass (*Leersia oryzoides*) dominated the lower areas, while goldenrod (*Solidago sp*.), grape (*Vitis sp*.) and hedge-nettle (*Stachys palustris*) were common in higher areas. The understory often includes seedlings of silver maple, and a lack of willow seedlings, indicating these taller willow areas will transition to a more typical floodplain forest in the future. In drier areas, goldenrod (*Solidago sp.),* grape (*Vitis sp*.), hedge-nettle (*Stachys palustris*), and verbena (*Verbena stricta*) were common. In lower areas, cutgrass (*Leerzia oryzoides*) and smartweeds (*Polygonum pennsylvanicum* and some *P. hydropiperoides*) were most common, although many other species were present.

- Floodplain forest is a common habitat-type at the SVAD-050, generally forming the border to all water bodies. Silver maple (*Acer saccharinum*) is the dominant tree species, although in a few locations, the forest is dominated by river birch (*Betula nigra*). Other common trees, especially in the maple-dominated areas, include green ash (*Fraxinus pennsylvannica*) and American elm (*Ulmus americana*). The maple-dominated areas comprise approximately 52 percent of the land area at the SVAD-050. The birch-dominated areas comprise approximately 5 percent of the SVAD-050 land area. In the deeper forest, nettle (*Urtica dioica*), smartweed *Polygonum spp,* especially *P. pennsylvanicum*), sedges (*Carex spp*.), and goldenrods (*Solidago sp*.) are common. In less dense and higher forest areas, button bush (*Cephalanthus occidentalis*) is abundant, as is cutgrass (*Leersia oryzoides*), goldenrod (*Solidago sp*.), hedge-nettle (*Stachys palustris*), young sandbar willow (*Salix interior*), and locust (both black [*Robina pseudoacacia*] and honey [*Gleditsia tricanthos*]). Many other species were noted, but were typically less common.

- Low floodplain meadow consists of basically open areas within or adjacent to the floodplain forest, mostly on downstream extensions of points or islands. It is limited to 11 fairly small locations, most of which were under water during the 2000 MWH field investigation. The description was based on what was apparently present on a 3-year-old color infrared photograph used in the field, and on remnants that could be seen. The low floodplain meadow areas comprise approximately 3 percent of the land area at the SVAD-050. The vegetation varies with ground elevation. In the lower portions, the

dominant plants include rice cutgrass (*Leersia oryzoides*), smartweeds (esp. *Polygonum pennsylvanicum*) and sedges (*Carex spp*.). In higher areas, cutgrass persists, but is joined by mixed forbs such as goldenrods (*Solidago spp*.) and hedge-nettle (*Stachys palustris*), and shrubs, especially buttonbush (*Cephalanthus occidentalis*). Sandbar willow (*Salix interior*) is also beginning to invade some of these areas.

- A few small areas on the SVAD-050 contain a drier, yet not upland (semi-upland), forest. These areas comprise approximately 2 percent of the land area at the SVAD-050. No one species dominated, but the most common were American elm (*Ulmus americana*), green ash (*Fraxinus pennsylvannica*), and honey locust (*Gleditsia tricanthos*), with other species (particularly oaks [*Quercus spp*.]) less common. There was usually a varied shrub component, but gray dogwood (*C. racemosa*) and grape (*Vitis sp*.) were most common. The understory was extremely varied from place to place, reflecting much of the diversity found in the adjacent grass/forb community, especially near the outer borders of these non-floodplain forests. No species could be considered dominant.

An open water vegetative community exists at the small pond in the northern SVAD-050 area. The pond was created by the excavation of borrow material for the demolition pad at SVAD-050. Common species noted along the pond's periphery include mud plantain (*Alisma subcordata*), spikerush (*Eleocharis spp*.), broad-leaf arrowhead (*Sagittaria latifolia*), milkweed (*Asclepias spp*.), cattails (*Typha latifolia*), and sedges. The shallow areas along the pond edge provide good foraging habitat for small mammals and birds.

1.7.6.3 Wetlands

There are approximately 5,712 acres of wetlands and 970 acres of open water and ponds located at SVDA. The wetlands consist primarily of floodplain forest. Floodplain forest is a common habitat-type at the SVAD-050, generally forming the border to all water bodies. Silver maple (*Acer saccharinum*) is clearly the dominant tree species, although in a few locations, the forest is dominated by river birch (*Betula nigra*). Other common trees, especially in the maple-dominated areas, include green ash (*Fraxinus pennsylvannica*) and American elm (*Ulmus americana*). The maple-dominated areas comprise approximately 52 percent of the land area at the SVAD-050. The birch-dominated areas comprise approximately 5 percent of the SVAD-050 land area. No significant impacts to wetlands will result from our field activities.

The aquatic ecosystem adjacent to the SVAD-050 includes portions of Crooked and Straight Sloughs, and backwater areas of the Mississippi River within Pool 13, south of L&D No. 12. In general, the backwater areas within Pool 13 consist of a series of shallow sloughs and oxbows with silt and sand substrates, often in layers at least 2 ft deep; they receive water from offshoot channels of the Mississippi River and from seasonal flooding. River stage is the dominant influence on aquatic habitat. The rising and falling water levels within the pool alter water depth, sediment transport, general water quality, and areal extent of potential aquatic habitat. Unlike the main river channel, water movement in the sloughs is usually minimal. The sloughs provide highly variable in stream habitat cover, including emergent and submergent macrophytes, submerged tree stumps and limbs, and root wads of fallen trees. Numerous aquatic flora and fauna inhabit the slough system, and the shallow areas provide important reproductive habitat for several fish species. No significant impacts to open water and the aquatic ecosystem will result from our field activities.

1.7.7 Cultural and Archaeological Resources

Section 106 of the National Historic Preservation Act (NHPA) requires federal agencies to take into consideration the potential effects of their actions on cultural resources that are eligible for, nominated to, and/or listed on the National Register of Historic Places (NRHP).

Archeological site files maintained by the Illinois State Historic Preservation Office (SHPO) and USACE provide evidence for prehistoric Native American and historic Euro-American sites at the SVDA. Previous cultural resources investigations indicate that human occupation may have been possible at the Depot dating to 10,000 years before present. Prehistoric sites are expected along terraces of the Mississippi and Apple Rivers.

Among the known prehistoric sites on the SVDA are Middle Woodland and Mississippian mounds containing Native American burials, as well as ceramic and/or lithic scatters dating from the Woodland, Late Woodland, Mississippian, and unknown cultural periods. Most portions of the depot have not been systematically investigated for prehistoric archeological sites.

SVDA contains many designated historic properties and potentially many historic archeological sites. European exploration of the Mississippi Valley began during the seventeenth century, although Colonial period sites have rarely been identified. Galena outcrops of Jo Daviess and Carroll Counties were mined for lead by Europeans and Native Americans since before the American Revolution, and early lead mining camps are most likely to have been located near the SVDA. The first permanent Euro-American settlement at Savanna was established during 1828. The first evidence for settlement on the SVDA is the James Beaty Old Stone House, built about 1850 and standing yet today. By the early twentieth century, historic maps indicated that at least 43 farmsteads were located on the SVDA property. All but the Beaty House were demolished or moved after the Army purchased the area during World War I. The extant Beaty House has been recommended as eligible for the NRHP. Other former nineteenth-century farmsteads might retain artifact assemblages and architectural features reflective of the Homestead Period (1824-1860) and/or Industrial Period (1860-Present) within northwestern Illinois, and might also meet criteria for eligibility to the NRHP. Because of the location of the project area on the Mississippi River floodplain, it is unlikely that homesteads were established in the area of the SVAD-050 kick-out/fragmentation range fan.

1.8 Previous Investigations

A variety of investigations at the OBG have been conducted over the years. The general intent and scope of the principal investigations are summarized below.

Installation Assessment (U.S. Army Toxic and Hazardous Materials Agency, 1979)
The U.S. Army Toxic and Hazardous Materials Agency (USATHAMA), now called the Army Environmental Command, assessed the past and current use of toxic and hazardous materials at SVDA and the potential for these substances to migrate from the Installation. The *Installation Assessment of Savanna Army Depot Activity, Record Evaluation Report No. 134* (USATHAMA, 1979) identified the OBG as Site 13 and as an area of concern at SVDA. Interviews with site personnel conducted by USATHAMA (1979) indicated that packaging materials for leaking mustard shells were burned at the southern burn pad inside the oval-shaped track between 1942 and 1945. The southern burn pad was designated by USATHAMA as Site 14. Subsequent

interviews conducted by Dames & Moore (1998) conflicted with previous information obtained regarding the handling of mustard waste at the OBG. The results of the interviews by Dames & Moore (1998) indicate three likely situations:

- A small number of ordnance items containing mustard were "experimentally" burned in pits behind the old office building, which reportedly was located south and west of the southern limit of the oval-shaped roadway that connects the various OBG burn pads.
- Burnable packaging materials, such as wood from carts used to transport mustard ordnance at SVDA, were burned at the pad originally identified by USATHAMA as Site 14 (which was excavated during the USACE (1996) demonstration project).
- Shells containing mustard agent were possibly buried at the OBG. Permanent markers in the general area where mustard ordnance burning is reported to have occurred identified geophysical signatures consistent with buried 155-mm shells.

Rapid Response Environmental Surveys (Environmental Science & Engineering, 1982)
In response to findings of the Installation Assessment (USATHAMA, 1979), Environmental Science & Engineering, Inc. (ESE, 1982) was contracted to perform preliminary environmental sampling and analysis at the OBG. The ESE sampling program conducted in 1982 consisted of collection and analysis of 5 soil samples and 18 sediment/surface water samples from a broad area around the site and installing and sampling a single monitoring well (301303). This limited monitoring confirmed the presence of site-related chemical constituents in the environment at the site. Subsurface geologic data were obtained during the installation of monitoring well 301303 at the site. Water level measurements obtained across the SVDA facility were used to assess the regional groundwater flow direction and hydraulic gradients.

Groundwater Contamination Survey (U.S. Army Environmental Hygiene Agency, 1988)
The U.S. Army Environmental Hygiene Agency (USAEHA) identified the OBG as a solid waste management unit (SWMU) in its report on the occurrence of SWMUs at SVDA (USAEHA, 1988). Based on interviews, USAEHA concluded that the OBG included multiple gravel burn pads that were used between approximately 1930 and 1985. A Dames & Moore (1998) air photographic survey revealed that none of the OBG burn pads existed until after 1939. USAEHA conducted no environmental sampling or analysis.

Environmental Monitoring Report (Hunter/ESE, 1989)
During the 1988 investigation, Hunter/ESE (1989) collected one confirmatory groundwater sample from the existing monitoring well and three additional surface water samples. The Hunter/ESE findings were consistent with the 1982 analytical results regarding site-related chemical constituents.

OBG RI/FS (Dames & Moore, 1998)
During 1992 and 1993, Dames & Moore conducted a RI/ FS field program. The scope of the 1992 field program was based on a review of historical site monitoring data (e.g., ESE, 1982 and Hunter/ESE, 1989). The 1993 RI/FS program further characterized areas of the OBG that were identified as potential contamination sources based on the 1992 data. The 1993 investigation was conducted to obtain additional information regarding the extent of soil and groundwater

contamination and to identify cleanup alternatives. The results of the investigations are summarized in Dames & Moore (1998).

Ordnance/Explosives Environmental Services Demonstration Project (USACE, 1996)
A demonstration project was conducted by Ordnance/Explosives Environmental Services (USACE, 1996) to evaluate potential operational problems associated with soil excavation and sifting as a remediation alternative to remove ordnance-related debris. Following UXO clearance, approximately 17,300 cubic yards (yd^3) of soil and debris from the eastern portion of the OBG and 3,000 yd^3 from the southern portion were successfully excavated, sifted, and stockpiled. Debris streams were identified and separated to facilitate disposal. Two rounds of soil sampling were conducted—one prior to excavation to assess health and safety requirements for workers, and the other from the stockpile of sifted soil to characterize the soil for final disposal. The sifted soil did not meet the USEPA's TCLP lead criteria, thus indicating the need for additional treatment to deal with the residual lead contamination.

After completion of the 1996 demonstration project, the treated soil was securely stockpiled at the OBG. The stockpile included approximately 20,000 yd^3 of soil in five piles. The stockpile area was referred to as the "soil retention area" (SRA). USACE conducted confirmation sampling and analysis to assess the potential requirements for additional soil remediation. The USACE sampling determined that one pile (6,300 yd^3) was characteristically hazardous for lead and the remaining four piles (13,700 yd^3) were nonhazardous.

Supplemental Sediment and Surface Water Investigation (Montgomery Watson, 1998)
Potential ecological risks were identified in a draft risk assessment (Dames & Moore, 1997) for the OBG. The ecological risk assessment (ERA) relied on data analyses obtained during field investigations conducted in 1992 and 1993. The results of the 1997 ERA were inconclusive because of the lack of an adequate background data set for the site and analytical detection limits that exceeded ecological risk assessment guidance values. Supplemental sediment, surface water, and soil sampling and soil/sediment toxicity testing was conducted in October 1997 by Montgomery Watson.

Stockpile Removal Action (Montgomery Watson, 2000)
In 1999, Montgomery Watson conducted a removal project for the SRA. The project consisted of the onsite stabilization of the characteristically hazardous soils and offsite disposal of the treated material as a special waste. All other piles were disposed of as nonhazardous soil.

Ecological Risk Assessment (Montgomery Watson Harza, 2003)
MWH conducted an investigation to support characterization of ecological risk at the OBG. The goal of the ERA was to assess contaminant toxicity and bioaccumulation that leads to adverse health effects at the OBG due to Army-related contamination in water, sediment, soil, and biota. The ERA examined native fish populations, populations of burrowing animals and wetland-dependent vertebrates, plant and invertebrate communities, and state or federally listed threatened and/or endangered species. The field investigation for the ERA included characterization of habitat, collection of samples to characterize levels of chemical exposure, and collection of samples to characterize levels of toxicity. Surface soil, sediment, surface water, and

ecological samples (fish tissue, plant, and flying insect) were collected. In addition, MWH conducted background sampling for fish, surface soil, sediment, and surface water.

Background and Groundwater Sampling (SAIC, 2002a)
In 1999 and 2000, SAIC conducted a groundwater monitoring program at the OBG to establish a current structural baseline (e.g., visually and physically inspect) and chemical baseline (i.e., sample) of groundwater monitoring wells to facilitate future site decision-making. Eleven of the 13 wells sampled by Dames & Moore were redeveloped and three rounds of sampling were conducted. In addition, from 1997 through 2001, SAIC collected background samples for soil, sediment, groundwater, and surface water to enhance the existing background data sets.

Human Health Risk Assessment (SAIC, 2002b)
In 2002, SAIC conducted two HHRAs at the OBG to evaluate risks to receptors under both current and future land uses. Because of analytical method detection limit issues associated with the older Dames & Moore data, one assessment included data collected by Dames & Moore, MWH, and SAIC, and a second assessment excluded the Dames & Moore data and used only the MWH and SAIC data. Both HHRAs evaluated risks to U.S. Fish and Wildlife Service (USFWS) workers, construction workers, recreational children and adults, and fishers.

Groundwater Sampling (USACE, 2003)
USACE conducted quarterly (August 2003) RCRA groundwater monitoring at SVAD-050 and the OBG including sampling for perchlorate in OBG monitoring wells. Groundwater samples were collected and analyzed from wells 301303 through 301309, 301311, 301402, 301404, and 301405 at the OBG, and wells MW01 to MW09 associated with SVAD-050.

Old Burning Ground Remedial Investigation Update (SAIC, 2004)
SAIC, under contract with USACE Louisville, prepared a Remedial Investigation (RI) report for the Old Burning Ground (OBG) at Savanna Army Depot Activity (SVDA), Savanna, Illinois. This has updated the report originally prepared by Dames & Moore in 1998 to include additional sampling conducted after 1996 with revised human health and ecological risk assessments.

Figure 1-1. Savanna Army Depot Location Map

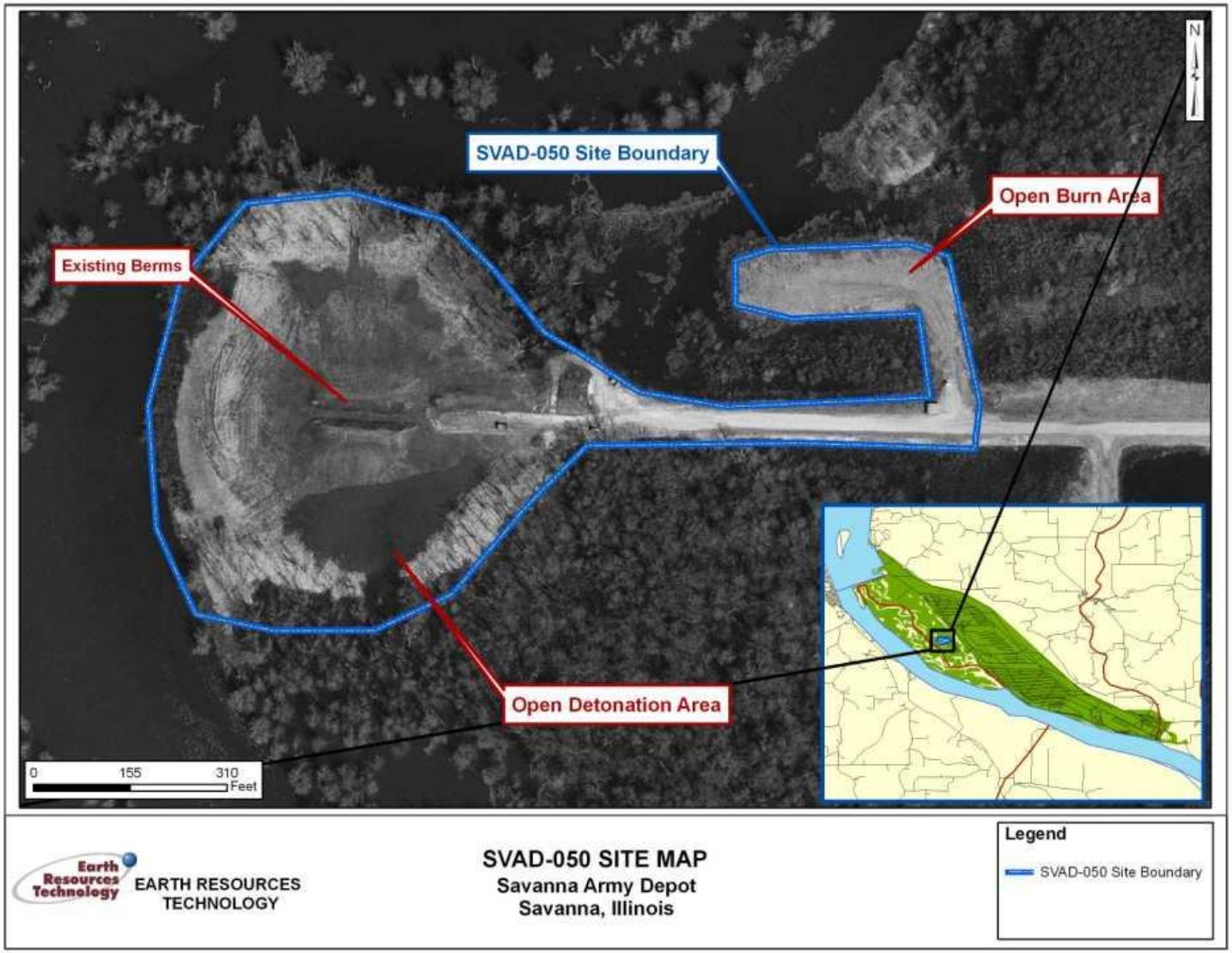

Figure 1-2. SVAD-050 Site Map

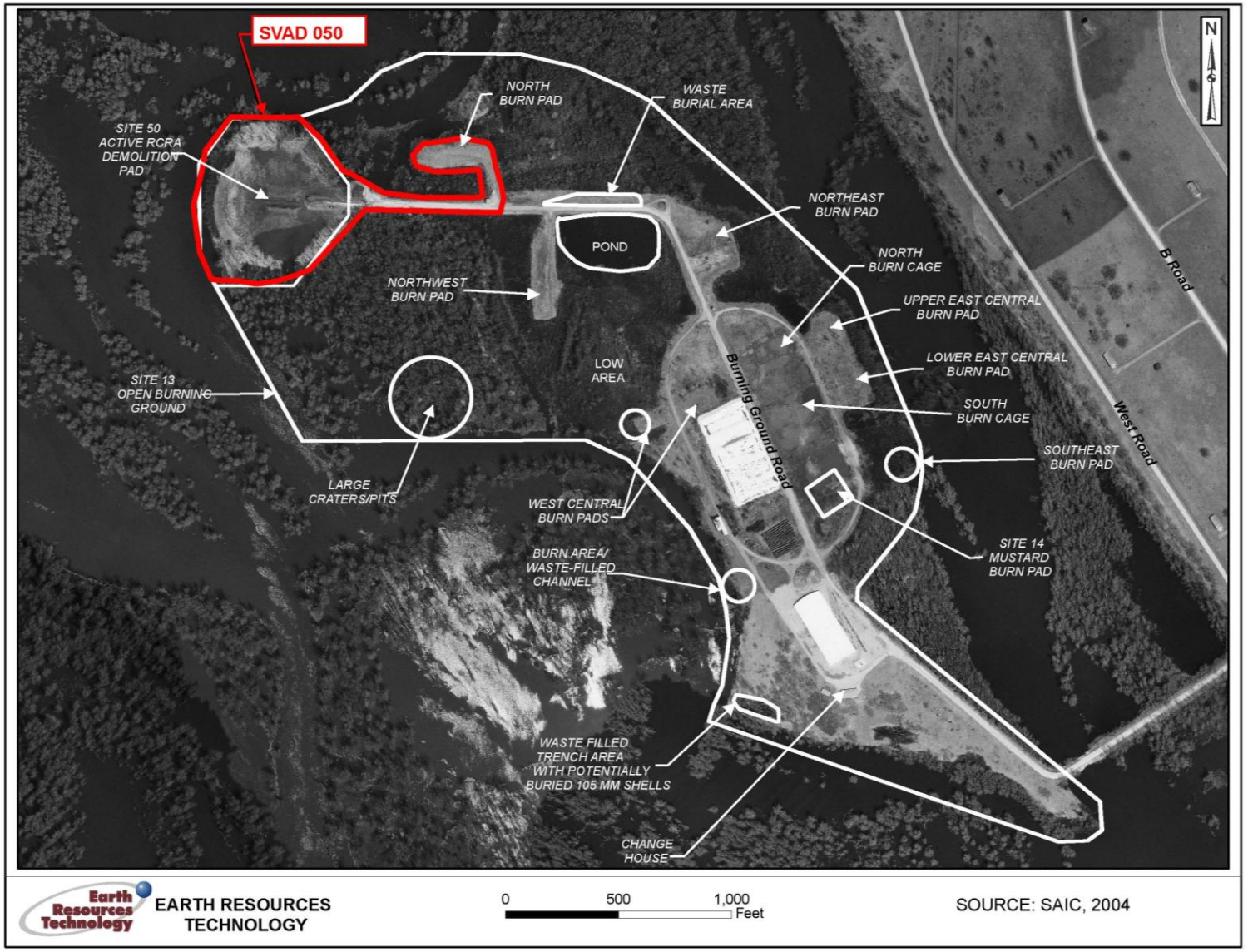

Figure 1-3. Layout of Old Burning Ground (SVAD-009-R-01)

2.0 PROJECT OBJECTVIES AND ORGANIZATION

2.1 Project Objectives

The overall objective of this project is to complete an RI/FS for SVAD-050 along with a Proposed Plan and ROD. The USACE Louisville District would prefer to integrate the actions of this RI/FS for the SVAD-050 area with the ongoing work at the OBG (SVAD-009-R-1). Since the SVAD-050 area is next to the OBG, the FS alternatives should match and complement the alternatives in development by SAIC for their FS on the OBG. Should the CERCLA process timelines for the SVAD-050 and OBG areas become synchronized, the U.S. Army may consider the integration of the sites and documents into one. Regardless of how the documents proceed, the USACE requires ERT to coordinate schedules, meetings, and actions with SAIC's efforts at the OBG. SAIC is ERT's primary subcontractor on this project, and will be responsible for performing the risk assessments and developing the FS for the SVAD-050 area.

To meet this objective, ERT has proposed an approach to adequately characterize the nature and extent of contamination at the site, collect information necessary for developing the human health and ecological risk assessments, and provide sufficient site data in order to develop and evaluate remedial alternatives. The following is a generalized approach for conducting this RI/FS. Details of specific activities are provided in the appropriate referenced sections or appendices of this work plan.

- Conducting all work in a safe manner for field personnel and authorized visitors (Appendix A).
- Surveying the site in order to develop grid system for conducting geophysical, MEC, and environmental sampling locations (Appendices B and C).
- Conducting limited vegetation clearance around the OD and OB sites (roughly 7 acres), sufficient to allow the geophysical, MEC, and environmental sampling to occur (Appendix C).
- Performing MEC surface clearance and demolition, if necessary (Appendix C).
- Conducting geophysical investigations (over roughly 7 acres) to identify subsurface anomalies that will aid in the location of test pits and surface/subsurface soil sampling (Appendix B).
- Performing field screening of surface soils across the entire site, using a 100 ft by 100 ft grid system (Appendix D).
- Excavating six test pits in areas where the geophysical survey identified a high incidence of anomalies. At least one test pit will be dug at each of the OB, and OD sites. A minimum of two soil samples will be collected for laboratory analysis at each test pit (Appendices C and D).
- Installing one additional groundwater monitoring well (Appendix D).
- Conducting environmental sampling of surface and subsurface soils, groundwater, surface water, and sediments (Appendix D).
- Analyzing environmental samples for potential contaminants (Appendix E).
- Conducting a human health and ecological risk assessment using existing analytical data as well as new analytical data collected during this study (Section 4.0).

- Developing and evaluating remedial alternatives to address any potential risks to human health and ecological receptors. If possible, these remedial alternatives will be part of the FS currently underway for the OBG (Section 5.0).
- Developing a Proposed Plan and ROD for the selected remedial alternative (Section 5.0).

2.2 Organization

The project team consists of the USACE - Louisville District, Savanna Army Depot (SVDA), ERT, and the following subcontractors: SAIC, CT Laboratories, Environmental Data Analysis, Randolph and Associates, Norwest Surveying Inc., and Aquadrill Inc. The overall project organization is provided in Figure 2-1. Other specialized vendors may be required during the project. Individuals assigned to the project team were selected to fulfill the requirements of project job descriptions as outlined in this section of the Work Plan.

2.2.1 U.S. Army Corps of Engineers, Louisville District

USACE - Louisville District is the, contracting, and funding agency for this project and is the t Project Technical Manager to provide technical oversight of this project. As the lead technical agency for this project, USACE responsibilities include procurement of service contracts and review and coordination of project plans and documents. USACE also provides technical expertise for MEC activities. As the Project Technical Manager, USACE is responsible for directing all aspects of the project.

2.2.2 Earth Resources Technology, Inc.

ERT will perform and manage the work described in this Work Plan (and accompanying Appendices) as appropriate and is responsible for all of the subcontractors.

Figure 2-1 shows the Project Organizational Chart. The following paragraphs describe the specific responsibilities of personnel assigned to the project team as they relate to the work to be conducted for the RI/FS. Resumes for additional key staff, not identified in the Final Quality Control Plan for this project (ERT, 2008), are provided in Appendix F.

Program Manager – Glenn Earhart
Glenn Earhart is responsible for contracting, oversees contract specifications to be included in the execution of the project task, and has ultimate responsibility for client satisfaction. Mr. Earhart has been the Program Manager for USACE Hazardous, Toxic, and Radioactive Waste (HTRW) and UXO projects for over 20 years.

Project Manager – Michael Dorman
Mr. Dorman is responsible for managing schedule changes and funding needs, providing appropriate resources for execution of the SOW, developing the project delivery team, and assisting in the design of the project with respect to overall objectives. As the Project Manager (PM), he is responsible for communicating with USACE through the USACE Project Technical Manager or the Contracting Officer's Representative (COR). He will execute all directions received from the USACE Contracting Officer, manage all aspects of the project, oversee the overall performance of all individuals on the project team, coordinate all contract and subcontract work, and resolve project issues.

The PM will also coordinate the preparation of the Work Plan and the implementation of on-site field activities. The PM will interface directly with subcontractors to keep them advised of the SOW, schedule, and budgets. The project manager is also responsible for ensuring that the subcontractor costs are within budget and that schedule commitments are achieved. The PM supervises the Site Manager and technical staff supporting the project. The PM is also responsible for the following:

- Preparing and submitting Purchase Orders;
- Resolving site project issues;
- Reviewing and approving Daily Activity Report;
- Maintaining an inventory and tracking of government furnished equipment;
- Procuring necessary equipment and supplies;
- Establishing, maintaining and tracking petty cash expenditures;
- Establishing and maintaining a Consumable Supplies Log;
- Reviewing and approving time sheets, expense reports, and travel order request;
- Submitting an Equipment Expense Report; and
- Ensuring that health and safety requirements are met.

Senior UXO Supervisor/Site Manager – Michael Garner
Mr. Garner will serve as the Site Manager and Senior UXO Supervisor (SUXOS). As the Site Manager, he is responsible for communicating with the ERT PM and field teams (UXO, geophysical, and environmental sampling), planning multiple field activities with multiple teams in order to utilize resources in an effective manner. As the SUXOS, Mr. Garner will also be responsible for:

- Identification of personnel and equipment requirements;
- Supervision of all daily field team activities;
- Early detection and identification of potential problem areas and institution of corrective measures;
- Overseeing project equipment maintenance program;
- Ensuring that all UXO and UXO-related material is properly identified, categorized and reported;
- Providing on-the-job training for selected UXO personnel who may be called upon to temporarily perform SUXOS duties during his absence from the site;
- Supervision of UXO Technicians; and
- Final Inspection and certification of Material Potentially Presenting an Explosive Hazard (MPPEH)

Project Geologist – Neil Jones
Mr. Jones will be the Project Geologist responsible for organizing day-to-day tasks of a multidisciplinary technical staff, promoting the efficient completion of tasks outlined in the scope of work and approved work plans. Mr. Jones will be the technical lead for the development of project planning documents, leading environmental sampling efforts, and coordinating activities with the other field teams (geophysical and UXO) and the Site Manager.

Project Geophysicist - Ji Ma
Mr. Ma will be the geophysicist of record and perform daily data review, processing, and dig-list picks. The Project Geophysicist will review all site-specific activities, progress of the survey, and identified problems. The Project Geophysicist will report all problems to the PM and will assist in correcting any problems as soon as possible.

Project Chemist – Sharif Mina
The project chemist will perform a quality control (QC) review of all analytical data and prepare a summary of the QC results. Mr. Mina will be responsible for generating reporting tables to be included in each report along with an interpretation of the data, if required, and cataloguing the data in Staged Electronic Data Deliverable (SEDD)/Automated Data Review (ADR) compatible format for electronic submittal.

UXO Safety Officer/UXO Quality Control Officer – George Payne
Due to the number of personnel on this site, the UXO Safety Officer (UXOSO) will be shared with the UXO Quality Control (UXOQC) Officer position. Mr. Payne will be responsible for implementing all Site Safety and Health Plan (SSHP) requirements, conducting on-site safety training and recommending changes to level of personal protection equipment (PPE) to the SUXOS as site conditions warrant. The UXOSO has Stop Work Authority for safety conditions. He will report all safety work stoppages immediately to the USACE on-site MEC Safety Specialist. The UXOSO evaluates and analyzes any potential safety problems, implements safety-related corrective actions, and maintains a Daily Safety Log. The UXOSO reports directly to the SUXOS and PM. The UXOSO will:

- Perform on-the-job training for selected UXO Technicians who may be called upon to temporarily perform the duties of UXOSO during his absence from the site, upon approval of the USACE MEC Safety Specialist; and
- Maintain daily liaison with the USACE on-site representative.

In the additional role of UXOQC, Mr. Payne will inspect/review all project operations, including explosives inventories, daily reports, time sheets and other documentation, and will inspect and approve each transect prior to turnover to the government representative. He maintains daily liaison with the USACE on-site representative.

UXO Technician III
This individual, who supervises a project team, shall be a graduate of a school listed in paragraph 10.2.a. or 10.2.b of Data Item Description (DID) Munitions Response (MR)-025.01. This individual shall have experience in MEC clearance operations and supervising personnel, and shall have at least ten years combined active duty military explosives and ordnance disposal

(EOD) and contractor UXO experience. This individual will be able to fully perform all functions enumerated for UXO Sweep Personnel, and UXO Technicians I and II. Specific duties of the UXO Technician III will include:

- Supervision of sweep teams during surface sweep operations;
- Supervision of demolition operations;
- Performance of explosive inventories;
- Supervision of equipment maintenance;
- Supervision/performance of ordnance-related scrap inspection;
- Determining whether MEC items are acceptable to move or need to be blown in place; and
- Supervision of UXO Technicians I and II.

UXO Technician II
A UXO Technician Level II shall be a graduate of a school listed in paragraph 10.2.a. or 10.2.b of DID MR-025.01. As an exception, a UXO Technician II may be a UXO Technician I with at least five years combined military EOD and contractor UXO experience. This individual will be able to fully perform all functions enumerated for UXO Sweep Personnel and UXO Technician I. The UXO Technician II's specific duties for this project will include:

- Performance of surface MEC clearance operations;
- Performance of demolition operations to destroy MEC;
- Assisting in ordnance-related scrap inspections under the Technician III's direction; and
- Performing equipment maintenance.

UXO Technician I
This individual shall be a graduate of a school listed in paragraph 10.2.c of DID MR- 025.01. The UXO Technician I's specific duties (under the supervision of a UXO Technician III or a UXO-qualified individual of higher rank than the UXO Technician III) for this project will include:

- Assisting in surface MEC clearance operations;
- Assisting in demolition operations to destroy MEC;
- Assisting in ordnance-related scrap inspections; and
- Assisting in performing equipment maintenance.

UXO Sweep Team
ERT will use one UXO Sweep Team. The UXO Sweep Team consists of seven individuals with at least one UXO Technician Level III, at least one UXO Technician II and up to five UXO Technician I/II sweepers. The UXO Sweep Team may be reduced as conditions dictate, provided that there are always at least two fully qualified UXO Technicians on the team (one of

which must be a UXO Technician III), and further provided that the team size does not exceed seven persons.

Demolition Team
ERT will not utilize a separate Demolition Team. The UXO Sweep Team will perform demolition duties as required. When actually performing demolition operations, the Demolition Team will consist of a minimum of one UXO Technician III and one UXO Technician II. The UXO Technician III will be in charge of, and oversee all operations of, the Team. Only UXO-qualified personnel will perform the actual explosive operations, however, they may be assisted by UXO Technician I.

2.2.3 Science Applications International Corporation (SAIC)

SAIC is an engineering and science firm with more than 2,300 engineering and environmental professionals. SAIC has completed more than 1,200 federal HTRW projects valued at more than $1.5B in the past 5 years. SAIC's strong dedication to customer commitment has distinguished SAIC as one of the Corps' most experienced and successful contractors, supporting 20 USACE Districts on 27 HTRW contracts during the past 25+ years. For the last 13 years, SAIC has provided proactive, responsive and comprehensive solutions to the Louisville District. These services have covered every facet of HTRW studies, designs and support of compliance and regulatory issues including site surveys, development of National Environmental Policy Act (NEPA) documentation, performance of risk assessments, environmental media sampling and lab analysis, and cost engineering.

SAIC is currently performing several environmental projects at SVDA including RI/FS of the OBG area, adjacent to the SVAD-050. As a subcontractor to ERT, SAIC will be responsible for performing a quality control review of the RI report, conducting the human health and ecological risk assessments, and preparing the FS (as part of a single document for the SVAD-050 and OBG). Keystaff from SAIC include Ms. Marcy Larriva, SAIC Project Manager; Ms. Linda Meredith, Human Health Risk Assessment Lead; Mr. Mike Barta, Ecological Risk Assessment Lead; and Ms. Vasu Peterson, Feasibility Study Lead.

2.2.4 CT Laboratories, Inc. (CT Laboratories)

CT Laboratories is validated by the USACE, U.S. Navy Facilities Engineering Service Center and maintains national accreditation through multiple National Environmental Laboratory Accreditation Program (NELAP) certifications. CT Laboratories holds an Illinois state certification. CT Laboratories will be performing all environmental sample analysis for both the quarterly groundwater monitoring and RI/FS components of this project. Mr. Pat Letterer will be the point of contact (POC) for CT Laboratories.

2.2.5 Environmental Data Services (EDS), Inc.

EDS will conduct the data validation of the laboratory analytical data, using the most recent USEPA Region II data validation Standard Operating Procedures (SOP). The EDS POC will be Mr. Dan Elwood.

2.2.6 Norwest Surveying Services, Inc.

Norwest Surveying Services, Inc. will provide surveying services. The POC at Norwest is Mr. William Holt, PLS.

2.2.7 Aquadrill, Inc.

Aquadrill, Inc. will provide services to install one groundwater monitoring well. The POC at Aquadrill is Mr. Jeff Joslyn.

2.2.8 Other Subcontractors

Additional subcontractors may be required during brush clearance, subsurface sample collection, investigation-derived waste (IDW) disposal, and well installation. Every effort will be made to work with local contractors when practicable.

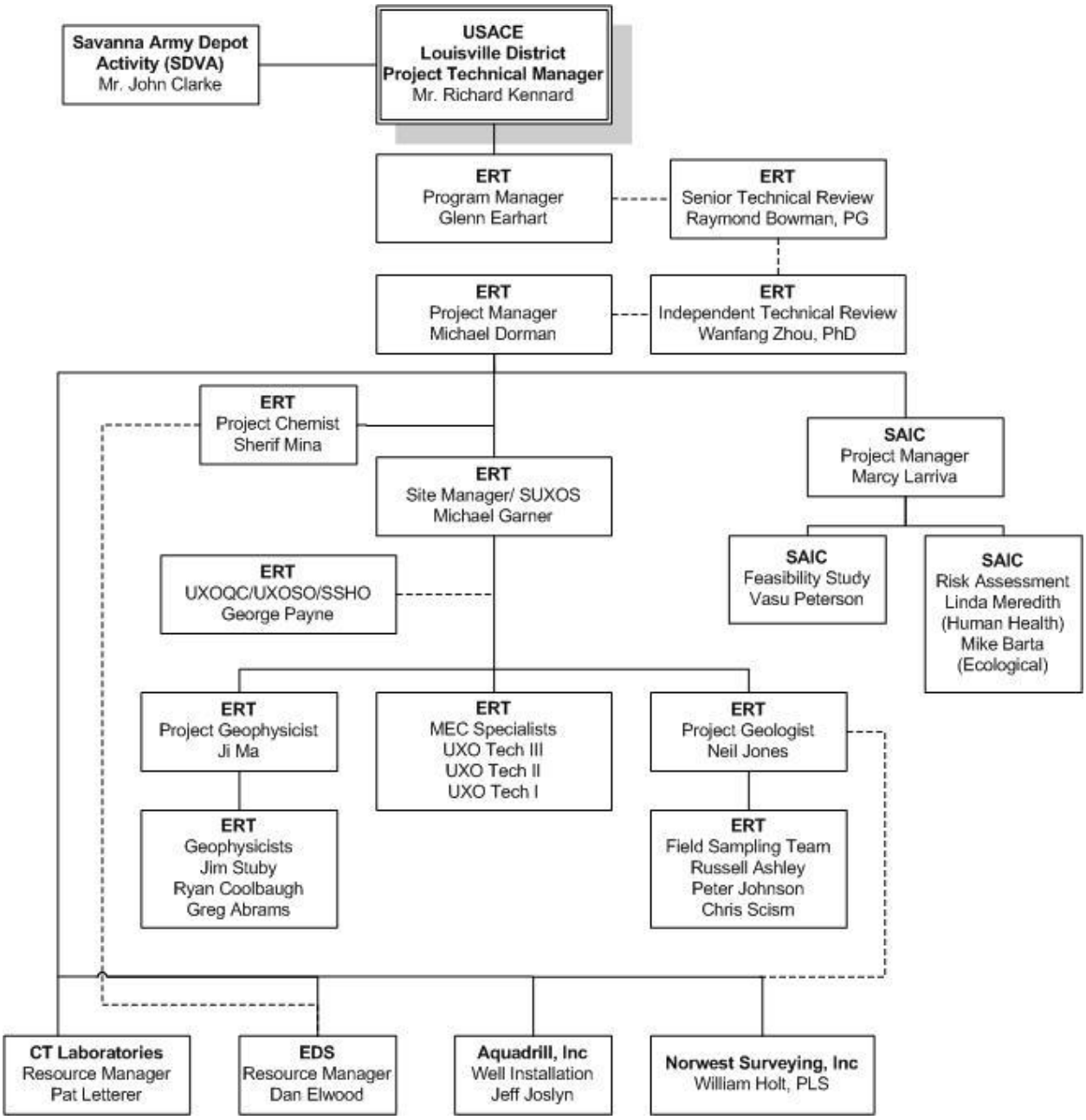

Figure 2-1. Project Organizational Chart

3.0 PROJECT MANAGEMENT AND REPORTING

3.1 Project Management

The Final Quality Control Plan (QCP) for the SVAD-050 RI/FS (ERT, 2008) provides an overview of ERT management structure and philosophy. The QCP also presents the project schedule for both the RCRA quarterly sampling effort and the RI/FS field sampling effort and report preparation. The following provides details regarding the management of various activities related to the field activities and report preparation specific to the RI/FS at SVAD-050.

3.2 Deliverables and Schedule

The major deliverables for the SVDA RI/FS include the following:

- Draft and Final Quality Control Plan;
- Draft and Final Plans for the RCRA Quarterly Groundwater Monitoring; including:
    - Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan),
    - Quality Assurance Project Plan, and
    - Site-Specific Safety and Health Plan
- Draft, Draft Final, and Final Work Plan for the RI/FS at SVAD-050, including :
    - Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan),
    - Site-Specific Safety and Health Plan,
    - Geophysical Investigation Plan, and
    - MEC Investigation Plan;
- Draft, Draft Final, and Final Remedial Investigation Report;
- Draft, Draft Final, and Final Feasibility Study;
- Draft, Draft Final, and Final Proposed Plan;
- Draft, Draft Final, and Final ROD (Record of Decision);
- Field Investigation Reports, including:
    - Weekly geophysical data;
    - MEC dig sheets;
    - Location maps and geophysical maps;
    - Daily QC Summary Reports (environmental sampling);
    - Electronic copy of the MC data ;
- Electronic copy of geographic information system (GIS) files related to this project;
- Monthly progress reports; and
- Meeting minutes.

The proposed project schedule has been provided in the Final Quality Control Plan (ERT, 2008).

3.3 Project Communications and Reporting

3.3.1 Communications

The ERT Project Manager will work in close communication with the USACE Louisville Project Technical Manager and the BRAC Environmental Coordinator (BEC) at SVDA to ensure that all project requirements are met, and to keep the USACE Louisville Project Technical Manager

informed of any technical or administrative issues that may impact the project schedule or budget. Any communication that has the potential to impact the project SOW, schedule, or budget will be confirmed via written correspondence between ERT and the USACE Louisville Project Technical Manager.

3.3.2 Field Reporting

While performing field work under this task order, ERT will prepare and submit weekly status reports in accordance with DID MR-085. In addition, monthly status reports will be provided (see Section 3.3.3).

3.3.3 Monthly Progress Reporting

Monthly project status reports that provide the status for the previous month's activities will be submitted to the USACE by the 15th of each month. The monthly report will contain summarized cost and performance information and statistical exposure data for program management purposes. The status reports will include the following information:

- Milestones Achieved (including deliverables submitted)
- Identify if any modifications to Delivery Orders were received
- Task order costs
- Identify project issues or risks
- Upcoming Activities/Milestones
- Identify if any key meetings were held, field work performed, etc.
- Percent complete for each task.

3.3.4 Project Billing

ERT's cost proposal provided the budgeted cost of work for each of the task elements as described in the previous section. This firm fixed price cost estimate will serve as the basis for ERT's payment plan, as it will provide discrete and measurable elements and activities associated with project progress. ERT will invoice on a monthly basis based on project percent complete, and in accordance with the accepted cost proposal, as presented below.

Task	Description	Billing
1	Work Plan Development	10% upon delivery of Final QCP, 50% upon delivery of Final RCRA plans; 100% upon delivery of Final RI/FS Plans
2	Quarterly Groundwater Sampling through 2008	35% upon completion of 3rd quarter sampling; 70% upon completion of 4th quarter sampling; 100% upon delivery of Final 2008 Sample Results Report
3	Quarterly Groundwater Sampling for 2009 (Optional)	20% upon completion of each quarterly sampling event; 100 percent upon delivery of Final 2008 Sample Results Report
4	Remedial Investigation Field Activities	See below
4.1	Installation and Sampling of New Groundwater Monitoring Well	100% upon completion of well and delivery of sampling results

Task	Description	Billing
4.2	Surveying and Vegetation Clearing	100% upon completion of surveying and vegetation clearing
4.3	MEC Surface Clearance and Test Pit Excavations	100% based on completion of MEC clearance and test pit excavations
4.4	Geophysical Prove Out (GPO) and Digital Geophysical Mapping (DGM)	10% upon delivery of Final GPO report; 100% based on completion of DGM
4.5	Phase I Soil Sampling – Field Screening	100% upon delivery of Phase I field screening results for surface soils
4.6	Phase II Soil Sampling	100% upon delivery of Phase II surface and subsurface soil sampling results
4.7	Surface Water and Sediment Sampling	100% upon delivery of surface water and sediment sampling results
5	RI Development	80% upon delivery of Draft RI, 90% upon delivery of Draft Final RI, and 100% based on delivery of Final RI
6	FS Development	80% upon delivery of Draft FS, 90% upon delivery of Draft Final FS, and 100% based on delivery of Final FS
7	Proposed Plan (PP) Development	80% upon delivery of Draft PP, 90% upon delivery of Draft Final PP, and 100% based on delivery of Final PP
8	Decision Document Development	80% upon delivery of Draft ROD, 90% upon delivery of Draft Final ROD, and 100% based on delivery of Final ROD
9	Project Management	4% of total PM amount each month.

Along with all contract-required documentation, each month the Project Manager will submit summary level information that supports each invoice submitted to the USACE - Louisville for review. This summary level information will be presented on Eng Form 93-1.

3.4 Management of Field Operations

This section describes the management of field operations during the sampling, geophysical and MEC investigation activities at SVDA, including site access and control, traffic control and parking, field team organization and responsibilities, and scheduling.

3.4.1 Site Access and Control

Access to the SVAD-050 and range fan area at the SDVA is controlled by gated access from a perimeter access road. Access to the site area from the Mississippi River is prevented by a buoy system around the perimeter of the area. In addition, access via the river would be difficult due to the shallow slough system around the site. All personnel engaged in site activities will attend a daily safety briefing given by the ERT UXOSO. All ERT and subcontractor personnel will receive site-specific training prior to starting work on the site. Areas associated with the MEC investigation will be closed to the public. Areas associated with the MEC disposal will be closed to the public and all other personnel not essential to the task being conducted in that area. UXO-qualified personnel and the geophysical teams will be considered essential personnel during intrusive activities. Unescorted access will be restricted to MEC cleared areas of the site. During the geophysical investigation, only minimal changes to the current accessibility of the

site will be required. Environmental sampling personnel will be considered nonessential personnel and their activities will be coordinated with the Exclusion Zone (EZ) requirements. Only personnel essential to the intrusive sampling process will be allowed inside the EZ.

Environmental sampling personnel will be escorted by UXO Technicians while conducting any intrusive (i.e., subsurface) sampling. Non-intrusive sampling will not require a UXO Technician escort after areas have been surface-cleared for MEC during previous phases of the project. However, should any field team member identify possible MEC in the field, work will be halted, the SUXOS will be notified, and work will not resume until the SUXOS takes necessary corrective action. Intrusive operations will be stopped if nonessential personnel or non-UXO qualified personnel are inside the EZ.

3.4.2 Field Office/Command Post

ERT will maintain a field office during this investigation, located at Building 1100 on SVDA. The field office will be used primarily as a staging area and to store equipment. ERT will use this location as a meeting point for morning meetings and daily briefings. The field office will not have telephone service, as mobile phone reception is adequate across the installation.

3.4.3 Traffic Control/Parking

Vehicle traffic and parking will generally be limited to existing roads and parking areas on the site. Only existing parking areas will be utilized. As needed, vehicles may need to travel off existing roads to move equipment and/or personnel to the investigation areas.

3.4.4 Communications

Telephone, fax, and Internet services may be available at the nearby hotel where the field crew will be staying. Field communication systems will include cellular telephones and two-way radios.

3.5 Data Management

Data will be collected from various operations on the work site and recorded on computer disks, in logbooks and on the various ERT, ATF, and DoD forms. The limits of the areas mapped and cleared, the type and location of MEC encountered, and the disposition of MEC will be recorded. The forms, logs and disks will remain in the ERT site office while portions of the data will be copied by the PM for inclusion in reports that need to be submitted.

4.0 REMEDIAL INVESTIGATION SCREENING AND RISK ASSESSMENT METHODOLOGY

The process for screening of the laboratory analytical data and conducting the human health and ecological risk assessments are detailed in SAIC's Data Collection Quality Assurance Plan-Volume I (1999), as well as subsequent negotiations as part of the comment resolution process. Some aspects of the risk assessment process have changed since 1999 and these changes are highlighted below:

- Screening of chemical and munitions constituent analytical data will be performed using the most recent IEPA Tiered Approach to Corrective Action Objectives (TACO) Tier I values and USEPA Regional Screening Levels (RSLs). Updates to Illinois groundwater quality standards and TACO objectives will be included as screening criteria, should they become available before the investigation is completed. Screening will first be performed on human health values, then against ecological values. Specifically, results from the various media will be compared to the following values:
    - Soil results will be screened against IEPA Tier 1 Soil Remediation Objectives for Residential Properties and USEPA Sept 2008 RSLs for Residential Soil.
    - Groundwater results will be screened against IEPA GW Quality Standards, USEPA Sept 2008 Tapwater RSLs, and USEPA MCLs.
    - Surface water results will be screened against IEPA SW Quality Standards for human health (or against Illinois' surface water criteria for chemicals not listed in the surface water quality standards) and USEPA Sept 2008 Tapwater RSLs.
    - Sediment will be considered to be screened against Illinois Sieved Stream Sediment Data, Illinois Tiered Approach for Evaluation and Remediation of Petroleum Product Releases to Sediments, the National Sediment Quality Survey, and/or USEPA Sept 2008 Residential Soil RSLs.
    - Ecological screening will be based upon USEPA Region 5 Ecological Screening Levels and Illinois Surface Water Quality Standards and/or criteria for Aquatic Life (chronic).
- The HHRA will follow the approved (by Army, USEPA, and IEPA) methodologies that SAIC has developed for other SVDA sites. New guidance to be incorporated into the SVAD-050 risk assessment will include Pro-UCL being used to calculate 95 percentile upper confidence limits (UCLs). In addition, exposure assumptions used in the risk assessment will be similar to or the same as those used in the OBG RI Update HHRA. Exposure assumptions will be updated as shown in Table 4-1.
    - Exposure scenarios will include soil and groundwater. Surface water and sediment exposures will be evaluated where there is realistic human exposure to these media.
    - The HHRA will evaluate four receptor scenarios: US Fish and Wildlife Service (USFWS) worker, construction worker, recreational receptors, and residents. The residential scenario is evaluated as an unrestricted scenario to determine if land use controls are necessary. The residential scenario includes a produce ingestion exposure pathway.

  - Risks will most likely be calculated for two exposure units (OD area and OB area), unless analytical results indicate both areas have similar chemical/munitions concentrations.
  - Chronic toxicity exposures will be evaluated.
  - A background comparison will be conducted using the bottomlands background data set used in the SVDA OBG RI Update (SAIC, 2004). The HHRA will be a stand-alone section and will include recommendations to be included in the RI.

- A screening ecological risk assessment (SERA) and a baseline ecological risk assessment (BERA) will be performed as part of the RI. The SERA will include the following components: a qualitative discussion of the habitat, PBT chemical and ecotoxicity screens, the quantitative risk characterization results (i.e., calculation of HQs) outlined in Section 4, a qualitative evaluation of ecoCOPCs that could not be evaluated quantitatively due to an absence of TRVs for one or more receptors, and a weight-of-evidence evaluation to help determine if the site recommendation should be NFA, further evaluation in a BERA, or whether inadequate information for decision-making is available.
  - The SERA and BERA will follow the approved (by Army, IEPA, and USEPA) methodologies and format that SAIC has developed for other SVDA sites. New guidance to be incorporated into the risk assessment will include Pro-UCL being used to calculate 95% UCLs.
  - The SERA and BERA will evaluate soil, surface water, and sediment exposures.
  - Groundwater concentrations will not be quantitatively evaluated because this medium is often inaccessible to ecological receptors. However, groundwater will be indirectly evaluated in situations where it discharges to surface water through the collection of surface water samples.
  - No additional bioassay sampling is currently planned.
  - The BERA will evaluate a subset of chemicals from the SERA; only those chemicals with hazard quotients (HQs) above one in the SERA will be evaluated in the BERA.
  - For all terrestrial receptors, risks will be calculated for two exposure units (OD area and OB area)
  - Only chronic toxicity exposures will be evaluated.
  - In general, SAIC will use the literature-based uptake factors that have been used previously at SVDA. If site-specific chemical data suggest a similarity in the types and magnitudes of detected concentrations between Site 50 and the OBG, then any site-specific uptake factors used previously at the OBG also would be used at Site 50. In such cases, justification will be provided in the RI Report.
  - For all aquatic receptors, risks will be calculated for one exposure unit assuming that the receptors can travel throughout this one unit (i.e., both the OB and OD area). Risks to reptiles/amphibians will also be evaluated (species to be determined).
  - Adjacent and neighboring site risks will be discussed qualitatively in the uncertainty section.

- For threatened and endangered species, additional considerations include:
    - The terrestrial plant TRVs are a mixture of Lowest Observable Effects Concentrations (LOECs) or No Observable Effects Concentrations (NOECs) depending on availability. When available, a NOEC will be used in the BERA to represent T&E terrestrial plant species.
    - No Observed Adverse Effects Level (NOAEL)-based TRVs will be used to evaluate potential for adverse effects for all of the wildlife species in the SERA. In general, both NOAEL- and Lowest Observed Adverse Effects Level (LOAEL)-based TRVs will be used in the BERA for wildlife receptors to provide a range from which to make risk management decisions.
    - To evaluate risks to T&E wildlife species potentially at Site 50 (which include the Upland Sandpiper [Bartramia longicauda] represented by the spotted sandpiper), only the NOAEL-based TRV will be used

Table 4-1. Exposure Assumptions for the Site 50 Human Health Risk Assessment

ROUTE ASSUMPTION	UNITS	FUTURE LAND USE											
		USFWS Worker		Construction Worker		Recreational				Residential			
						Child		Adult		Child		Adult	
		RME		RME		RME		RME		RME		RME	
General													
Body weight	kg	70	b	70	b	15	b	70	b	15	b	70	b
Exposure duration	years	25	b	3	j	6	b	30	b	6	b	30	b
Averaging time - noncancer	days	9,125	b	1,095	b	2,190	b	10,950	b	2,190	b	10,950	b
Averaging time - cancer	days	25,550	b	25,550	b	25,550	b	25,550	b	25,550	b	25,550	b
Soil Ingestion													
Ingestion rate	mg/day	100	h	330	f	200	b	100	b	200	b	100	b
Exposure frequency	days/year	104	k	50	j	52	l	52	l	350	b	350	b
Conversion factor	kg/mg	1E-06	-	1E-06	-	1E-06	-	1E-06	-	1E-06	-	1E-06	-
Soil Dermal Contact													
Skin surface area available Soil to skin adherence factor	cm2/day mg/cm2	3,300 0.2	g g	3,300 0.3	g f	2,800 0.2	g g	5,700 0.07	g g	2,800 0.2	g g	5,700 0.07	g g
Dermal absorption factor	unitless	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-
GI absorption factor	unitless	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-
Exposure frequency	days/year	104	k	50	j	52	l	52	l	350	b	350	b
Conversion factor	kg/mg	1E-06	-	1E-06	-	1E-06	-	1E-06	-	1E-06	-	1E-06	-
Particulate Inhalation													
Exposure Time	hours/day	8	h	8	h	8	h	8	h	24	h	24	h
Particulate emission factor	m3/kg	1.36E+09	f	1.36E+09	f	1.36E+09	f	1.36E+09	f	1.36E+09	f	1.36E+09	f
Volatilization factor	m3/kg	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-
Exposure frequency	days/year	104	k	50	j	52	l	52	l	350	b	350	b
Groundwater Ingestion													
Ingestion rate	L/day									1	h	2	a
Exposure frequency	days/year	not evaluated		not evaluated		not evaluated				350	b	350	b
Conversion factor	mg/µg									1E-03	-	1E-03	-
Groundwater Dermal Contact													
Skin surface area available	cm2/day									6,600	g	18,000	g
Dermal permeability constant	cm/hour									chemical specific		chemical specific	

Table 4-1. Exposure Assumptions for the Site 50 Human Health Risk Assessment

ROUTE ASSUMPTION	UNITS	FUTURE LAND USE									
		USFWS Worker		Construction Worker		Recreational				Residential			
						Child		Adult		Child		Adult	
		RME		RME		RME		RME		RME		RME	
Exposure time	hour/day	not evaluated		not evaluated		not evaluated				1	g	0.58	g
Exposure frequency	days/year									350	b	350	b
Conversion factor	L/cm3 and mg/µg									1E-06	-	1E-06	-
Groundwater Inhalation (Volatiles)													
Volatilization factor	L/m3									0.5	c	0.5	c
Exposure time	hour/day	not evaluated		not evaluated		not evaluated				24	h,i	24	h,i
Exposure frequency	days/year									350	b	350	b
Conversion factor	mg/µg									1E-03	-	1E-03	-
Sediment Ingestion (wading)													
Ingestion rate	mg/day	100	n			200	n	100	n	200	n	100	n
Exposure frequency	days/year	52	k	not evaluated		26	l	26	l	52	m	52	m
Conversion factor	kg/mg	1E-06	-			1E-06	-	1E-06	-	1E-06	-	1E-06	-
Sediment Dermal Contact (wading)													
Skin surface area available	cm2/day	3,300	n			2,800	n	5,700	n	2,800	n	5,700	n
Sediment to skin adherence factor	mg/cm2	0.2	n	not evaluated		0.2	n	0.07	n	0.2	n	0.07	n
Dermal absorption factor	unitless	chemical specific	n			chemical specific	n	chemical specific	n	chemical specific	n	chemical specific	n
Exposure frequency	days/year	52	k			26	l	26	l	52	m	52	m
Conversion factor	kg/mg	1E-06	-			1E-06	-	1E-06	-	1E-06	-	1E-06	-
Surface Water Dermal Contact (wading)													
Skin surface area available	cm2	3,300	n			2,800	n	5,700	n	2,800	n	5,700	n
Dermal permeability constant	cm/hour	chemical specific	-			chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-
GI absorption factor	unitless	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-	chemical specific	-
Exposure time	hour/day	1	o	not evaluated		1	o	1	o	1	o	1	o
Exposure frequency	days/year	52	k			26	l	26	l	52	m	52	m
Conversion factor	L/cm3	1E-06	-			1E-06	-	1E-06	-	1E-06	-	1E-06	-

Table 4-1. Exposure Assumptions for the Site 50 Human Health Risk Assessment

ROUTE ASSUMPTION	UNITS	FUTURE LAND USE							
		USFWS Worker	Construction Worker	Recreational		Residential			
				Child	Adult	Child		Adult	
		RME	RME	RME	RME	RME		RME	
	and mg/µg								
Produce									
Consumption rate (belowground)	kg/day	not evaluated	not evaluated	not evaluated		0.00023	e	0.00014	e
Consumption rate (above ground protected)	kg/day					0.0015	e	0.00061	e
Consumption rate (above ground unprotected)	kg/day					0.00077	e	0.00032	e
Bioconcentration factor	unitless					chemical specific	-	chemical specific	-
Exposure frequency	unitless					350	b	350	b

a - USEPA 1989, *Risk Assessment Guidance for Superfund (RAGS): Volume I, Human Health Evaluation Manual, Part A*
b - USEPA 1991a, *Human Health Evaluation Manual, Supplemental Guidance: Standard Default Exposures*
c - USEPA 1991b, *Risk Assessment Guidance for Superfund (RAGS): Volume I, Human Health Evaluation Manual, Part B;*
d - USEPA 1997, *Exposure Factors Handbook*
e - USEPA 1998, *Human Health Risk Assessment Protocol for Hazardous Waste Combustion Facilities*
f - USEPA 2002, *Supplemental Guidance for Developing Soil Screening Levels at Superfund Sites*
g - USEPA 2004, *Risk Assessment Guidance for Superfund Volume I: Human Health Evaluation manual (Part E, Supplemental Guidance for Dermal Risk Assessment)*
h - USEPA 2008, Regional Screening Levels for Chemical Contaminants at Superfund Sites, Generic Tables and User's Guide
i - ORNL 2008, Risk Assessment Information System
j - The construction worker works 5 days per week for 10 weeks over a duration of 3 years
k - The USFWS worker exposure frequency for soils corresponds to 4 days per week for 6 months of the year or 2 days per week for the entire year; the exposure frequency for sediments and surface water corresponds to 2 days per week for 6 months or 1 day per week for the entire year
l - The recreational receptor exposure frequency for soils corresponds to 2 days per week for 6 months of the year or 1 day per week for the entire year; the exposure frequency for sediments and surface water corresponds to 1 day per week for 6 months of the year
m - The resident exposure frequency for sediments and surface water corresponds to 2 days per week for 6 months of the year or 1 day per week for the entire year
n - In the absence of information for sediments, the parameters for the soil ingestion and soil dermal contact pathways will be used in the analagous sediment pathways.
o - Professional judgment for this activity

5.0 FEASIBILITY STUDY, PROPOSED PLAN, AND RECORD OF DECISION

5.1 Feasibility Study

The process for developing and evaluating remedial alternatives for the SVAD-050 RI/FS are detailed in SAIC's Data Collection Quality Assurance Plan-Volume I (SAIC, 1999). There are no overall changes to this methodology since originally developed in 1999. Some clarifications are included below:

- Remedial alternatives will be screened for applicability and applicable alternatives will be evaluated in detail for soil and groundwater including no further action.
- The same remedial alternatives addressed in the OBG FS will be used for the SVAD-050 FS.

5.2 Proposed Plan

The Preferred Alternative for the site will be presented to the public in the Proposed Plan. The Proposed Plan briefly summarizes the alternatives studied in the RI/FS, highlighting the key factors that lead to identifying the Preferred Alternative. The Proposed Plan, as well as the RI/FS and the other information that forms the basis for the USACE's response selection, will be made available for public comment in the Administrative Record file.

5.3 Record of Decision

Following receipt of public comments and any final comments, USACE will document the remedy selection decision in a Record of Decision and submit the proposed remedial action alternative(s) to US EPA and IEPA. The ROD documents the remedial action plan for a site or operable unit and serves the following three basic functions:

- It certifies that the remedy selection process is carried out in accordance with CERCLA and the NCP;
- It describes the technical parameters of the remedy, specifying the methods selected to protect human health and the environment including treatment, engineering, and institutional control components, as well as cleanup levels; and
- It provides the public with a consolidated summary of information about the site and the chosen remedy, including the rationale behind the selection.

While the ROD should provide a comprehensive description of site conditions, the scope of the action, the Selected Remedy, cleanup levels, and the reason for selecting the remedy, it is only one part of the Administrative Record file, which contains the full details of site characterization, alternatives evaluation, and remedy selection.

6.0 ENVIRONMENTAL PROTECTION PLAN

6.1 Site Characterization and Mitigation Approach

The Environmental Protection Plan (per DID MR-005-12.01) describes ERT's site-specific methods and procedures to minimize pollution, protect and preserve natural resources, and control noise and dust within the established standards. ERT's goal is to complete the scope of work on schedule and without a safety or environmental incident. ERT will achieve this goal through careful planning, training, supervision and quality control related to all aspects of the project.

At the completion of the project the project site will be restored to its original condition as appropriate.

6.2 Existing Conditions

Sensitive biological resources, including federal and state listed plant and animal species, sensitive plant communities, and archeological sites are known to occur within the SVDA boundaries. Several of these resources are also known to occur within the investigation sectors, and therefore could be impacted by the field investigations. Protection of these resources is required and will be accomplished during all phases of the investigation.

6.3 Potential Environmental Impact

6.3.1 Potential Environmental Impact

The SVAD-050 was used for burning and demolition of ordnance material, which caused physical alterations to the bottomlands and surrounding slough area. Deactivated ordnance material either was dumped onto the ground surface and covered with fill, or buried in trenches. The backfilling of waste material disturbed the open terrestrial habitat and the riparian zone along the backwater slough; as a result, there is no natural stage environment at the site. A large portion of the SVAD-050 is flooded for several weeks each spring. The flooding heavily influences vegetation and the types of available fauna habitats. Variation in plant communities appeared to be completely determined by elapsed time since disturbance by factors such as flooding and ground elevation. Large numbers of new species were noted between the 2003 MWH survey and the 1994 Dames & Moore survey. These observations, along with changes evident from 1997 color infrared photography, indicate that some areas of the SVAD-050, particularly the grass/forb mix, are undergoing a fairly rapid succession (MWH, 2003).

6.3.2 SVAD-050 Activities

In general, past SVAD-050 activities have apparently disturbed these communities to varying degrees. While various stages of succession were observed, no noted disturbances in community structure or distribution suggest causes other than the profound physical disturbance of the ground (Montgomery Watson, 1998). In addition, no protected plant species were found in SVAD-050 during the 1992 or 2000 surveys.

The Depot contains potential cultural resources dating from before and during the military phase of facility development. Previously recorded prehistoric and historic site locations within

SVAD-050 are on file with the Illinois SHPO and USACE. ERT does not anticipate any risk to cultural resources associated with the work at the SVAD-050 site. If a potential adverse effect is revealed, ERT will work with the USACE to develop an acceptable approach for addressing significant cultural resources located within the SVDA.

Archeological issues and concerns for the SVAD-050 kick-out/fragmentation range fan are focused on potential resources that may be affected by the removal of surface or subsurface MEC. Because of the proximity to slough area and the remoteness of the site, ERT does not anticipate any risk to archeological resources associated with surface or subsurface cleared items in the SVAD-050 kick-out/fragmentation range fan. However, given the cultural historical importance of the Mississippi River valley, the potential for the discovery of prehistoric sites is anticipated, including the presence of Native American burials. ERT anticipates that this action will leave all such sites undisturbed. Historic period cultural resources that predate the military use of the Depot would most likely relate to former farmsteads, and might include structural features and associated artifacts from dwellings, outbuildings, mines, railroads, and possibly cemeteries. It is anticipated that historic period refuse disposal areas (middens) could be located within the SVDA. Middens might contain metal objects related to activities such as farming, railroad construction, construction, architectural remains, and domestic activities that could give signals during geophysical actions comparable to MEC-related items. Impacts to cultural resources, including ground disturbing effects of vehicular traffic and geophysical investigations, are anticipated to be minimal. Blast in place activities will be localized.

6.3.3 Tree and Shrub Removal and Protection

Tree and brush clearing will be completed only where geophysical surveying will be conducted and, if, necessary, on the grids selected for UXO Investigation. Trees 4 inches or greater diameter will not be removed unless approved by a Government representative. Trees not scheduled for removal will be clearly marked with colored flagging in the field by ERT personnel. ERT will provide protective measures for trees that are not to be removed during clearing. To minimize disturbance to sensitive vegetation, the following mitigation measures will be implemented:

- Herbaceous vegetation less than four inches tall will not be cut.
- Removal of woody vegetation will be limited to specimens with a diameter at breast height (dbh) less than 4 inches. Care will be taken to ensure that no woody vegetation with a dbh greater than four inches is removed. Measurements will be taken if dbh is in question.
- Root mass will be not disturbed in forested areas.
- If necessary, vegetation will be removed with a clean cut at ground level to minimize trip hazards.
- All cleared areas will be allowed to re-vegetate naturally.
- If necessary, clearing of lower branches will be limited to 6 ft above the ground.

6.3.4 Existing Waste Disposal Sites

The OBG includes an old burn area for explosives (Site 13) and a burn site for 12 - 155 mm H-filled projectiles (Site 14). There are multiple burn pads at Site 13 that were used between approximately 1930 and 1985 (SAIC, 2004). Site 14 consists of a single burn site that was used

between 1942 and 1945 to burn 12 - 155 mm H-filled projectiles (SAIC, 2004). SVAD-050 was a RCRA Interim Status facility. Open detonation occurred, as needed, for detonating UXO identified during environmental investigations. Based on aerial photography analysis (Dames & Moore, 1998), there are no active waste disposal sites surrounding the SVAD-050 area. .

6.4 Mitigating Procedures

6.4.1 Manifesting, Transportation and Disposal of Waste

The most significant IDW during this project is the ordnance-related scrap recovered during the surface clearance. Any item expected to contain a potential explosive hazard will be detonated in place to remediate the hazard and therefore ensure that the item is acceptable for transportation and disposal. Qualified UXO technicians will perform a 100 percent inspection of ordnance-related scrap and complete a DD Form 1348-1A, in accordance with the Defense Utilization and Disposal Manual DOD 4160.21-M. The scrap certification will contain the statement:

> "This certifies and verifies that the ammunition, explosives, and dangerous articles residue, range residue, MEC scrap and/or Explosive Contaminated property listed have been 100 percent inspected and to the best of our knowledge and belief, are inert and/or free of explosives or related material."

Individual MEC items will be field screened for explosives and handled appropriately; either as non-hazardous metal scrap or as metal scrap with explosive residue. All certified scrap and residue will be turned over to a scrap dealer. The scrap dealer will provide a statement that the material will be processed through a smelter, and therefore making it unrecognizable. Other non-hazardous solid waste generated as a result of activities associated with this project will be transported to and disposed of in a municipal landfill. No tracking or manifesting of this type of waste is required.

Additional IDW consists of rinsate from equipment decontamination procedures, purge and development water from the newly installed monitoring well (MW-11), and soil cuttings from the installation of MW-11. All spent cleaning solutions, MW-11 purge/development water, and soil cuttings shall be individually drummed for disposal per ERT SOP #06-4, IDW Management (see Appendix E, Attachment 3). Rinsate, monitoring well IDW, and soil cuttings will be expected to be characterized using TCLP.

6.4.2 Burning Activities

SVAD-050 was previously used as an OB/OD area; however the open burn area of the site has not been used since 1985. No burning activities are anticipated during the conduct of the RI/FS activities at the site, including the MEC investigation (see Appendix C).

6.4.3 Dust and Emission Control

Heavy equipment will not generally be required for this task order. Light trucks and all-terrain utility vehicles may be used on a limited basis during this project. Brush clearing will be performed using a Brush Cutter attached to a Bobcat skid steer loader. ERT will limit the operation of trucks and will, to the extent practicable, avoid creating high traffic areas. Lighter

vehicles may used to collect scrap and consolidate it for pick-up by a heavier vehicle. All vehicles will be operated at low rates of speed to reduce the dust emitted. Dust emissions will be controlled through the use of administrative controls, such as speed limits and varied traffic patterns.

6.4.4 Spill Control and Prevention

Vehicles designed to travel on roads will be fueled at commercial filling stations that are designed to prevent and control potential spills. Fuel for use in utility vehicles will be transported and dispensed from fuel cans designed to reduce the potential for spills. Fuel will be transported in small containers (five gallons or less) and kept in a designated "fuel truck" equipped with fire extinguishers and spill kits. Fueling will be conducted in areas designated by the SUXOS. Spill kits will be maintained in the same vicinity. Fuel will not be stored on site and will be delivered in small quantities, only as needed. No overnight fuel storage is anticipated. Fuel will be transported in an unoccupied vehicle compartment (i.e., the bed of a pickup truck). The fuel containers will be filled at commercial filling stations.

6.4.5 Storage Areas and Temporary Facilities

ERT anticipates leasing existing warehouse/office space available at SVDA for limited storage of equipment and sampling supplies. No storage or temporary facilities will be constructed during the RI/FS activities.

6.4.6 Access Routes

ERT does not anticipate the need to construct access routes. The site will be accessed from existing roads and off-road routes designated by the SUXOS. Off-road routes will be modified and/or varied to prevent erosion and/or dust problems.

6.4.7 Control of Water Run-On and Run-Off

Because of the proximity of the SVAD-050 site to the Mississippi River slough network, the crater/soil disturbance associated with items that are blown in place, will be backfilled and returned to its original condition as quickly as possible to reduce the exposure of soil to run-on/run-off. These disturbances will be localized and there will be no significant erosion or sediment control concerns.

6.4.8 Decontamination and Disposal of Equipment

At the completion of the project and during the project, as required, equipment will be cleaned to remove dirt and dust. Methods will include both dry decontamination and wet decontamination. Wet methods will be performed in an approved location or wash facility. Disposable equipment will not be decontaminated. It will be transferred to an appropriate disposal facility.

6.4.9 Minimizing Areas of Disturbance

ERT will only work in areas defined in the SOW and will avoid activities outside of these areas. The work required by the scope is generally non-intrusive with the exception of test pits, subsurface soil sampling, and potentially localized crater/soil disturbance associated with items that are blown in place. These activities will cause minimal disturbance in the work area.

6.5 Post-activity Clean-up

After completing this task, the SUXOS or site geophysicist will be responsible for ensuring that all areas included in the scope are properly cleaned up. No debris will be left as a result of ERT's activities on the site. Facilities used by ERT will be cleaned and restored to their original conditions.

7.0 IDW MANAGEMENT PLAN

7.1 Approach

The IDW Management Plan (per USEPA Technical Memorandum 9345.3-03FS) describes ERT's site-specific methods and procedures to manage the waste materials that will be derived from the RI/FS at SVAD-050, in order to ensure protection of human health and the environment and comply with regulatory requirements that are applicable or relevant and appropriate requirements (ARARs). These include regulations under RCRA, the Clean Water Act (CWA), the Clean Air Act (CAA), the Toxic Substances Control Act (TSCA), and other State environmental laws.

The procedures in this RI/FS will be performed in such a way that IDW generation will be minimal to reduce the need for special storage and disposal requirements that may result in substantial additional costs yet provide little or no reduction in site risks relative to the final remedial actions.

Complementary information regarding the management of IDW generated during proposed RI/FS activities at SVAD-050 is presented in section 6.0 of this WP, and various sections of the QAPP.

7.2 IDW Management

7.2.1 General

IDW will include:

- Rinsate from equipment decontamination procedures
- Purge water from newly installed groundwater monitoring well (MW-11)
- Ordnance-related scrap recovered during the surface clearance
    - non-hazardous metal scrap
    - metal scrap with explosive residue
- Soil cuttings from the installation of MW-11
- Disposable PPE

*Though soil from test pit excavations is also considered IDW, it will not be addressed further in this section. Test pit soil will not be characterized for disposal and will instead be spread around the excavation pits.

As IDW is generated, it will be stored onsite in a designated area and remain in that location until characterization is complete. IDW will be placed in new or reconditioned, Department of Transportation (DOT)-approved 55-gallon drums. Drums will be in good condition and suitable for transportation. IDW drums will be placed in a configuration that allows room for inspections, operations and maintenance, and handling. Each drum will be labeled with the following information: contents, name of generator, and date.

IDW will be disposed of promptly after characterization is performed. Once the IDW is characterized, a determination will be made as to the proper management. Each container will

be referenced to a particular set of analytical (sample) data based on sample identification. Before receipt of data, all IDW will be characterized based on site knowledge, field observations, and field analytical data. All classification/characterization activities will be conducted when analytical data are received. IDW containers will be routinely inspected to ensure that all containers remain in serviceable condition and to ensure good housekeeping practices. Container inspections will be used to identify any problems associated with drum usage, such as bulging, leaking, or improper/missing labels. Any problems will be addressed immediately upon discovery

7.2.2 Liquid IDW Management

IDW rinsate liquids from equipment decontamination procedures and purge water from development of MW-11 will be collected at the various points of generation and deposited on-site in staged 55-gallon steel drums according to ERT SOP #06-4, *IDW Management* (presented in Attachment 3 of the QAPP). Environmental (GW, SW, soil, sediment) sample results will be sent to the off-site water treatment facility in advance of the IDW for determination of proper disposal method. Upon request of the disposal facility, the liquid IDW may also be sampled and characterized further for TCLP analyses and RCRA characteristics (see IDW Reference and Limits Table, QAPP Attachment 1). If other analyses become necessary, the parameters and methods will be provided as an attachment to the QAPP.

Liquid IDW sampling methods will be in accordance with ERT SOP #06-14, *Drum Handling and Sampling*, and will utilize dedicated dippers and glass tube samplers. Dippers will be used to collect samples from the surface of the liquid, if the waste is homogeneous. Otherwise, glass tube samplers will be used to collect a full-depth liquid sample from the drum(s). Grab sampling is the preferred method, though compositing may be necessary if the liquid IDW is to be characterized by TCLP methods and more than 1 drum of liquid is generated.

7.2.3 Solid IDW Management

All scrap munitions debris (MD) uncovered during the MEC Survey will be collected, screened for residual explosives with Expray® or DropEx® according to ERT SOP #06-10, Explosives Field Screening, and containerized in steel 55-gallon drums according to ERT SOP #06-4. If other analyses become necessary, the parameters and methods will be provided as an attachment to the QAPP. All MD scrap will be disposed of off-site. Scrap that tests negative for explosives will be handled and disposed of as non-hazardous waste and scrap that tests positive for explosives will be handled and disposed of as having explosives residue (see section 6.4.1 of WP, above).

Soil cuttings recovered during the installation of MW-11 will be containerized in a 55-gallon drum. Waste soil will be sampled by advancing a hand auger through the containerized volume of soil (not just grabbing a sample off the top) and characterized for TCLP analyses and RCRA characteristics. If other analyses become necessary, the parameters and methods will be provided as an attachment to the QAPP.

The containerized MD wastes and any soil determined to be hazardous will be transported by a subcontractor to the proper off-site disposal facility(s).

7.2.4 Disposable PPE Management

Disposable PPE (e.g., gloves, shoe covers) used in field activities will be containerized in steel 55-gallon drums. If characterization results from MD and/or liquid IDW indicate contamination with RCRA hazardous waste, the disposable PPE will be disposed of in compliance with RCRA Subtitle C requirements. Otherwise, it will be disposed of as regular, industrial refuse.

7.2.5 IDW Transporters and Disposal Facilities

The containerized MD and liquid wastes will be transported by a subcontractor to their off-site disposal facility. Alter Trading Company (ATC) will be responsible for transporting and disposing of all MD that leaves SVAD-050.

Alter Trading Company
Dubuque Processing yard
Dubuque IA 52002
(563) 583-1731

ATC will also be instrumental in the transport and disposal other solid and liquid wastes.

In addition to complying with requirements of Federal and State laws, all off-site disposal of wastes will comply with CERCLA section 121(d)(3) and the CERCLA Off-Site Policy (OSWER Directive No. 9834.11 (November 13, 1987)).

7.2.6 IDW Record Keeping

If hazardous wastes are sent off site, ERT will comply with both administrative and substantive elements of the RCRA generator requirements of 40 CFR Part 262.

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APPENDIX A
SITE-SPECIFIC SAFETY AND HEALTH PLAN

FINAL
SITE-SPECIFIC SAFETY AND HEALTH PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION (OB/OD) AREA, SITE SVAD-050, SAVANNA ARMY DEPOT ACTIVITY SAVANNA, ILLINOIS

Prepared for

U.S Army Corps of Engineers
Louisville District

Contract Number: W912QR-08-D-0012
Delivery Order 0001

Prepared by

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September 2009

FINAL
SITE-SPECIFIC HEALTH AND SAFETY PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION (OB/OD) AREA, SITE SVAD-050,
SAVANNA ARMY DEPOT ACTIVITY
SAVANNA, ILLINOIS

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APPROVED BY

9/2/09

Michael Dorman Date
Project Manager

5/20/09

Program Manager Date

COMPLETION OF SENIOR TECHNICAL REVIEW

This document has been produced within the framework of the Earth Resources Technology, Inc. (ERT) and ISO Approved 9001:2000 quality management systems certification. As such, a senior technical review, as defined in the project Quality Control Plan, has been conducted. This included review of the overall design addressed within the document, proposed or utilized technologies and alternatives and their applications with respect to project objectives, and Department of the Army policy and regulatory guidance and constraints.

Raymond J. Bowman 12/19/2008

Raymond Bowman Date

Senior Technical Reviewer

COMPLETION OF INDEPENDENT TECHNICAL REVIEW

This document has been produced within the framework of ERT's total quality management system and ISO 9001:2000 certification. As such, an independent technical review, appropriate to the level of risk and complexity inherent in the project as defined in the project Quality Control Plan, has been conducted. This included review of assumptions (methods, procedures, and material used in analyses), alternatives evaluated; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets the project objectives. Comments and concerns resulting from review of the document have been addressed and corrected as necessary.

Wanfang Zhou 5/20/09

Dr. Wanfang Zhou Date

Independent Technical Reviewer

CERTIFICATION

This Site-Specific Safety and Health Plan (SSHP) has been prepared under the supervision of, and has been reviewed by, a Certified Industrial Hygienist (CIH) certified by the American Board of Industrial Hygiene (ABIH).

12/1/08

Dewey Cubit, CIH Date

ABIHNo.CP1254

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ACRONYM LIST

ABIH	American Board of Industrial Hygiene
AHA	Activity Hazard Analysis
ANSI	American National Standards Institute
CFR	Code of Federal Regulations
CGI	Combustible gas Indicator
CIH	Certified Industrial Hygienist
CNS	Central Nervous System
CPR	Cardiopulmonary Resuscitation
CRZ	Contamination reduction Zone
dBA	Decibels on the A-Weighted Scale
DNT	dinitrotoluene
DoD	Department of Defense
EOD	Explosive Ordnance and Disposal
ERT	Earth Resources Technology, Inc
EZ	Exclusion Zone
FS	Feasibility Study
FSP	Field Sampling Plan
GPR	Ground Penetrating Radar
GPS	Global Positioning System
HAZWOPER	Hazardous Waste Operations and Emergency Response
HR	Heart Rate
HSM	Health and Safety Manager
HTRW	Hazardous, Toxic, Radioactive Waste
IFP	Interface Probe
kV	Kilovolt
LEL	Lower Explosive Limit
MEC	Munitions and Explosive of Concern
MPPEH	Materials Potentially Presenting an Explosive Hazard
MSDS	Material Safety Data Sheets
NIOSH	National Institute for Occupational Safety and Health
NRR	Noise Reduction Rating
OB	Open Burn
OD	Open Detonation
OE	Ordnance and Explosives
OSHA	Occupational Safety and Health Administration
OVM	Organic Vapor Meter
PDS	Personnel Decontamination Stations
PETN	Pentaerythritol tetranitrate
PID	Photo Ionization Detector
PM	Project Manager
PPE	Personal Protective Equipment
PPM	Part Per Million
QAPP	Quality Assurance Project Plan

QCP	Quality Control Plan
RCRA	Resource Conservation and Restoration Act
RDX	Cyclotrimethylenetrinitramine
RI	Remedial Investigation
RTS	Robotic Total Station
SLM	Sound Level Meter
SOP	Standard Operating Procedure
SOW	Scope of Work
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SUXOS	Senior Unexploded Ordnance Supervisor
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
TACO	Tiered Approach to Corrective Action Objectives
TCE	Trichloroethene
TNT	Trinitrotoluene
TWA	Time Weighted Average
USACE	US Army Corp of Engineers
UXO	Unexploded Ordnance
VOC	Volatile Organic Compound
WBGT	Wet-bulb Globe Temperature
XRF	X-Ray Fluorescence

1.0 INTRODUCTION

Earth Resources Technology, Inc. (ERT) has been contracted by the U.S. Army Corps of Engineers (USACE), Louisville District to conduct a Remedial Investigation (RI) and Feasibility Study (FS) at the Savanna Army Depot Activity (SVDA) Site 50. As part of the planning process, a Site-specific Health and Safety Plan (SSHP) was developed which establishes procedures to protect employees of ERT, subcontractors, USACE, and site visitors from potential safety and health hazards resulting from activities conducted during this project. This SSHP has been developed in accordance with requirements set forth in:

- 29 Code of Federal Regulations (CFR) 1910.120 - Occupational Safety and Health Agency (OSHA) Hazardous Waste and Emergency Response (HAZWOPER) Standard;
- 29 CFR 1926 - OSHA Safety and Health Regulations for Construction;
- ERT's Corporate Health and Safety Program;
- USACE *Safety and Health Requirements Manual*, Engineering Manual (EM) 385-1-1 (2003); and
- USACE *Unexploded Ordnance (UXO) Support During Hazardous, Toxic, and Radioactive Waste (HTRW) and Construction Activities*, Engineering Pamphlet (EP) 75-1-2 (2004a)

This SSHP has been prepared in order to provide safe procedures and practices for personnel performing site work. This SSHP was developed using contract information provided by the USACE, Louisville District and will refer to the requirements discussed in OSHA regulations, USACE EM 385-1-1 and EP 75-1-2, and ERT's Corporate Health and Safety Program. All of these documents will be kept onsite (or will be readily available), and are incorporated into the SSHP by reference. The SSHP will also be transmitted to all site workers and subcontractors.

1.1 Site History

SVDA is a military facility operated by the Department of the Army since 1918. Historically SVDA has been utilized for storage, assembly and demolition of various explosives, such as trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), and pentaerythritol tetranitrate (PETN), by detonation. The SVDA Site 50 (SVAD-050) area is comprised of approximately 7 acres and consists of three focus areas, an open burn (OB) site, an open detonation (OD) site, and the roads between the two sites. The OB pads were used to burn unstable propellants and the OD grounds were used to detonate obsolete projectiles and ordnance. The OB sites consisted of metal pans elevated above concrete pads by steel beams used to dispose of propellant. The propellants were destroyed by burning and the residual ashes were containerized on-site for disposal. The OB pads were not used except for emergencies and the most recent burn event occurred in 1985. The OD area consisted of two long earthen berms where pits were located, three pits per berm. Obsolete projectiles were destroyed energetically with a series of "shots." After the series of "shots" the surrounding area was searched for unexploded material. Materials that were not sufficiently destroyed were either collected and prepared for the next series of shots or detonated in place. The OD grounds were most recently used in the disposal of unexploded ordnance (UXO) generated during SVDA restoration activities. An interim Resource Conservation and Restoration Act (RCRA) status allowed demilitarization operations to continue

in the OD until October 2008. For more details on the history and usage of SVAD-050 area including site maps, see Section 1 of the primary Work Plan document.

1.2 Project Description

The overall RI project objectives for SVAD-050 Area include the following:

- Evaluate the nature and extent of contamination at SVAD-050 by determining whether existing contaminants of potential concern exceed U.S. EPA Screening Levels or Illinois Tiered Approach to Corrective Action Objectives (TACO) health-based guidance values, thereby warranting further evaluation (potential risk assessment and/or removal action).
- Assess potential ecological and/or human health risks from constituents of potential concern.

If potential risks are present, accumulate data sufficient to quantitatively delineate the impacted area and provide an estimate of the total volume of impacted media. This information will be reported in an RI and will be essential for the development of a future FS.

The key elements of the work to be conducted at SVAD-050 that are addressed in this SSHP include:

- Providing UXO technician escort to field crews (sampling, surveying, and geophysical teams);
- Conducting vegetation clearing;
- Performing surface clearance of Munitions and Explosives of Concern (MEC) on up to 7 acres of the site.
- Digging test pits in areas that have suspected anomalies.
- Performing down hole clearance for groundwater well installation and subsurface soil sampling

Field Work will be performed in accordance with the guidelines contained in the Sampling and Analysis Plan (SAP) and SSHP. The SAP contains the Field Sampling Plan (FSP), which should be reviewed with the Quality Assurance Project Plan (QAPP). Analytical data will be evaluated against applicable standards as discussed in the QAPP.

2.0 ORGANIZATION OF PERSONNEL

Role	Person	Contact Information
Program Manager	Glenn Earhart	(256) 217-8991
Project Manager (PM)	Michael Dorman	(301) 323-1401
Project CIH	Dewy Cubit	(301) 696-8501
Site Manager/SUXOS	Michael Garner	(919) 889-5588
Site Safety and Health Officer (SSHO)	George Payne	(301) 323-1421
USACE Project Technical Manager	Richard Kennard	(502) 315-6323

2.1 Program Manager

The Program Manager is responsible for contracting, oversees contract specifications to be included in the execution of the project task, and has ultimate responsibility for client satisfaction.

2.2 Project Manager

The Project Manager (PM) will be responsible for the overall direction, implementation, and enforcement of health and safety requirements. Other responsibilities include:

- Ensure that the project is being performed in a manner consistent with ERT's Corporate Health and Safety Program.
- Ensure that the SSHP is prepared and approved.
- Provide the Site Health and Safety Officer (SSHO) with project information related to health and safety matters and the development of the SSHP.
- Monitor compliance with the SSHP by ERT and subcontractor personnel.
- Ensure adequate resources are provided to the health and safety staff, so that they may carry out their duties.
- Ensure that all ERT and subcontractor personnel designated to work at the project sites are qualified according to ERT's medical surveillance and training requirements.
- Maintain communication with the USACE Authorized Representative.

2.3 Project Certified Industrial Hygienist

The Project CIH or his/her designee's responsibilities are listed below.

- Provides for the approval of the SSHP and works with the project team to ensure implementation of the SSHP;
- Ensures that all health and safety activities identified in the SSHP are conducted and/or implemented;
- Serves as the primary contact to review health and safety matters that may arise;
- Approves revised or new safety protocols for field operations;
- Coordinates revisions of this SSHP with field personnel;
- Oversees the investigation of major accidents/incidents;

2.4 Site Manager/Senior Unexploded Ordnance Supervisor

The Site Manager/Senior UXO Supervisor (SUXOS) reports directly to the PM and is responsible for the following items:

- Senior UXO technical leadership;
- Planning, coordinating and supervising all on-site UXO activities;
- Preparation of standard operating procedures for MEC operations ensuring compliance with Department of Defense (DoD), Army, and National Guard directives, as well as local, state, and Federal statutes and codes;
- Certification of Materials Presenting a Potential Explosive Hazard (MPPEH) and/or range scrap as ready for turn-in or disposal in accordance with current policies;
- Directing the MEC-related work performed under the scope of work;
- Supervising multiple project teams which may be performing MEC removals and related activities (e.g., vegetative clearance, land surveying, reconnaissance, and classification of MEC items, military explosives, demolition materials, and locating surface and subsurface MEC);
- Destroying MEC by burning or detonation, and/or transporting and storing MEC materials;
- Safe operation of all heavy equipment;
- Compliance with the SSHP;
- Ensuring all work is performed in compliance with the site plans; Federal, state, and local regulations governing the project; and all aspects of the scope of work;
- Authorizing the stoppage of work if unacceptable safety and health conditions exist, and taking necessary action to re-establish and maintain safe working conditions;
- Coordinating daily with the USACE Safety Officer(s) on planned field activities;
- Reviewing health and safety documentation to ensure compliance with this SSHP;
- Work with the Site Safety and Health Officer (SSHO) to identify, evaluate, and control hazards;
- Initiating the evacuation of the work site when needed, communicating with offsite emergency responders, and coordinating activities of onsite and offsite emergency responders; and
- Determine if hazardous conditions are adequately alleviated prior to allowing resumption of work operations after an emergency.

2.5 Site Safety and Health Officer

The SSHO will be onsite throughout the project and will be responsible for daily compliance with site safety and health requirements. The SSHO will have the following responsibilities:

- Ensure all site activities are performed in a manner consistent with ERT's Corporate Health and Safety Program and the SSHP.
- Interface with the Project CIH about on-site implementation of the SSHP.
- Direct daily health and safety activities onsite.

- In conjunction with the PM, ensure that all of ERT's personnel and subcontractors designated to work at the project sites are qualified according to ERT's medical surveillance and training requirements.
- Report all incidents, accidents, and near misses to the PM, Site Manager and to the USACE Authorized Representative.
- Maintain health and safety equipment onsite.
- Inspect ongoing activities, and report any health and safety deficiencies to the Site Manager and Project Manager.
- Accompany or maintain communication with each work crew.
- Perform site monitoring to ensure that site personnel are adequately protected.
- Conduct initial site-specific safety training and regular safety briefings for site personnel.
- Conduct periodic inspections for compliance with the SSHP;
- Monitor compliance with this SSHP; and
- Maintain Accident/Incident Report Forms

In addition, the SSHO will be a certified unexploded ordnance (UXO) specialist and perform the duties of the UXO Safety Officer (UXOSO) which include:

- Authorize the stoppage of work if in the UXOSO's professional opinion the hazard or condition poses an imminent danger to site personnel, the public or the environment, and take necessary action to re-establish and maintain safe working conditions;
- Implementing the approved MEC safety program in compliance with applicable (DoD), Federal, state, and local statutes and codes;
- Analyzing MEC, operational risks, hazards, and safety requirements;
- Establishing and ensuring compliance with all project-specific safety requirements for MEC activities;
- Enforcing personnel limits and safety exclusion zones for MEC clearance operations, MEC transportation, storage, and destruction;
- Conduct safety inspections to ensure compliance with MEC safety codes; and
- Coordinate daily with the USACE Safety Officer(s) on planned field activities.

The SSHO will have the authority to take the following actions:

- Stop site activities if an "imminently dangerous" situation exists. The emergency situation will be immediately reviewed with the PM, the Site Manager and the USACE Authorized Representative.
- Direct personnel to change a work practice if it is determined to be hazardous to the health and safety of site personnel.
- Temporarily suspend an individual from site activities for infractions of the SSHP, pending discussion with the Site and Project Managers.

George Payne will be designated as the Site Health and Safety Officer (SSHO) and UXOSO for all field tasks. The primary responsibility will be to ensure that all requirements of this SSHP are

fully enforced for all field activities under their direction. The primary purpose of this position is to provide UXO anomaly avoidance support for team personnel during field activities. The SSHO has the authority to stop work for health and safety concerns should the need arise.

2.6 Unexploded Ordnance Technicians

UXO Technicians are responsible for the direct supervision and safety of the personnel under their control, and they report to the Senior UXO Supervisor (SUXOS). They are responsible for ensuring that all work accomplished and all personnel comply with the site-specific work plan, SSHP, and all applicable Federal, state, and local regulations. The SUXOS may conduct additional safety meetings and training as needed. They report all safety violations and accidents/injuries to the UXOSO.

2.6.1 Unexploded Ordnance Technician III

This individual, who supervises a project team, shall be a graduate of a school listed in Table 1. This individual shall have experience in MEC clearance operations and supervising personnel, and shall have at least 10 years combined active duty military Explosive Ordnance and Disposal (EOD) and contractor UXO experience. A UXO Technician III is responsible for supervising and performing on-site disposal of MEC; preparing explosives storage plans in accordance with all applicable guidance; preparing required MEC administrative reports; preparing SOPs for on-site MEC operations; performing risk hazard analyses; conducting daily site safety briefings; and supervising the conduct of all on-site evaluations directly related to MEC activities.

2.6.2 Unexploded Ordnance Technician II

This individual shall be a graduate of a school listed in Table 1. As an exception, a UXO Technician II may be an UXO Technician I with at least 5 years combined military EOD and contractor UXO experience. A UXO Technician II is responsible for properly storing MEC material in accordance with applicable guidance; identifying fuzes and determining fuze condition; determining a magnetic azimuth using current navigational and/or locating equipment; performing field expedient identification procedures to identify explosives contaminated soil; preparing an on-site holding area for MEC material; and operating modes of transportation for transporting MEC material, when appropriate.

2.6.3 Unexploded Ordnance Technician I

This individual shall be a graduate of a course listed in Table 1. A UXO Technician I can advance to the UXO Technician II category after 5 years combined active duty military EOD and contractor UXO experience. This individual assists fully qualified personnel (UXO Technician II and above) in the following functions: conducting reconnaissance and classification of UXO and other MEC materials; identifying all munitions including bombs and bomb fuzes, guided missiles, projectiles and projectile fuses, rockets and rocket fuzes, land mines and associated components, pyrotechnics items, military explosives and demolition materials, grenades and grenade fuzes, and sub munitions; locating subsurface UXO using military and civilian magnetometers and related equipment; performing excavation procedures on subsurface UXO; locating surface UXO by visual means; transporting UXO and demolition materials; preparing firing systems, both electric and non-electric, for destruction operations; operating Personnel Decontamination Stations (PDS); inspecting salvaged MEC related material and erection of UXO related protective works; and donning and doffing personal protective equipment (PPE).

2.7 Field Geophysicist

The field geophysicist will have a minimum of 5 years of experience in field geophysics and demonstrate the ability to perform the following tasks:

- Operate the Juniper Allegro data collector provided with the Geonics EM61;
- Set up and operate all geophysical equipment being used during the data collection portion of this project;
- Operate Robotic Total Station (RTS); and
- Operate DAT61 software provided with the Geonics EM61.

2.8 Environmental Field Technicians

Environmental field technicians will be responsible for collecting environmental samples, including soil samples, groundwater samples, Expray testing, and wipe samples. Field Geophysicists may serve as Environmental Field Technicians. All Field Technicians will have OSHA 40-hour training. Field Technicians will perform the following tasks:

- Receive and establish accountability for project equipment;
- Organize support facilities and test communication equipment;
- Operate pumps for groundwater sampling;
- Operate Field X-Ray Fluorescence (XRF) unit;
- Operate RTS unit;
- Collect soil samples;
- Perform explosive residue screening tests using Expray field test kits; and
- Fill out Chain of Custody forms and ship environmental samples to laboratory.

2.9 Other Field Personnel

ERT field personnel will be identified as this project proceeds. Their responsibilities are listed below.

- Comply with rules, regulations, and procedures as set forth in this SSHP and any revisions;
- Maintain up-to-date knowledge of the information, instructions, and emergency response actions maintained in the SSHP;
- Report any unsafe or potentially hazardous conditions to the SSHO or Project Manager;
- Prevent admittance to work sites by unauthorized personnel; and
- Inspect all tools and equipment utilized by ERT for safe operation and condition, including PPE, daily prior to use.

2.10 Visitors

Authorized site visitors, defined as anyone who is not a regular project worker, such as contracting agency and other Federal or local agency personnel, may visit the site per the project-specification, but will be responsible for the following items:

- Receiving the site hazard and safety instructions from the UXOSO;

- Reviewing and complying with the essential elements of the SSHP;
- Using their own, or provided, PPE to enter regulated work areas when such controls are required for entry;
- Reporting any observed unsafe act and/or condition at, or affecting, the work site to the PM

Any official visitor who seeks entry into an Exclusion Zone (EZ) or Contamination Reduction Zone (CRZ) will present documentation of health and safety training in compliance with OSHA 29 CFR 1910.120 and medical surveillance examination and certification, and respirator fit testing when site conditions warrant. If the official visitor does not present or have documentation of health and safety training all UXO/HTRW operations will cease until he/she has left the EZ. The visitor, while inside the CRZ/EZ, will be escorted by a qualified site worker at all times.

While UXO operations are in progress, only those personnel necessary for the operation will be allowed within the exclusion zone. If nonessential personnel enter the area, all UXO operations will cease and these individuals will be escorted by qualified UXO personnel. A Visitors Log (included in Attachment 1) documenting name of the visitor and time of the visit will always be maintained at the site.

3.0 ERT HEALTH AND SAFETY PROGRAM

3.1 General Safety and Health Training

ERT requires all field sampling team members to participate in a minimum of 40 hours of OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) safety training sessions outlined in 29 CFR 1910.120. The subjects addressed include: regulatory concepts, toxicology, first aid, material safety data sheets (MSDS), field monitoring, personal protection, site-entry procedures, sampling and decontamination, documentation, and emergency response. Annual 8-hour refresher courses are also required for each field sampling and geophysics team member prior to any field work.

3.2 UXO Personnel Training

UXO personnel assigned to positions UXO Technician I, UXO Technician II, UXO Technician III, UXO Safety Officer, UXO Quality Control Specialist, and Senior UXO Supervisor, shall have the requirements shown in Table 3-1.

Table 3-1. UXO Personnel Training and Experience Hierarchy

Position Description	Training Required (Notes 1, 2, & 3)	Minimum Years of EOD/UXO Experience	Special Requirements
UXO Safety Officer	1, 2, or 3	8 Years	Experience in all phases of UXO remediation and applicable safety standards.
UXO Quality Control Specialist	1, 2, or 3	8 Years	Experience in all phases of UXO remediation and the transportation, handling and storage of ordnance and explosives materials.
Senior UXO Supervisor	1, 2, or 3	10 Years	Significant experience in all aspects of UXO remediation. Five years experience in supervisory positions.
UXO Technician III	1, 2, or 3	8 Years	Prior military EOD and/or commercial UXO experience.
UXO Technician II	1, 2 or 3	3 years	Prior military EOD experience Experience in UXO remediation/range clearance operations. Plus specific project/ordnance training.
UXO Technician I	1, 2, or 3	0	Successfully completed DOD certified course of instruction appropriate to this skill level

Notes:

1. Graduate of the Army Bomb Disposal School at Aberdeen, MD.
2. Graduate of the Naval EOD School
3. Graduate of a Department of Defense certified UXO Training Program
4. This is the minimum experience requirement for designation. This is not an automatic designation, but reserved for those who have demonstrated the requisite knowledge, maturity, judgment and are recommended by the contractor for recognition at the specified skill level.

3.3 Site-Specific Training

In addition, the following training requirements will also be satisfied:

- Employees must be provided site-specific safety and health indoctrination and training to enable them to perform their work in a safe manner;
- This training will be documented as required by EM 385-1-1 Section 01.B.02;
- Field personnel assigned to the project will be informed of, and trained on, the content and application of the SSHP;
- Field personnel will review the SSHP and sign a SSHP Compliance Agreement upon completing this training (see Attachment 1);
- OE identification, safety and health hazards, operating procedures, and emergency response procedures; and
- All personnel entering the project must attend a Safety Briefing prior to entry.

All personnel assigned to the intrusive investigation teams will attend a site-specific orientation. The purpose of this orientation will be to review site-specific and emergency response procedures. The topics to be covered during the orientation are listed below. Attendance sheets will be used to document completion of each orientation session.

3.4 Medical Surveillance

All ERT personnel who will be performing activities at SVAD-050 will have undergone an extensive medical examination by a board certified occupational physician. The purpose of this evaluation program is listed below.

- Assess the health status of personnel prior to work, including ability to use respiratory protection;
- Evaluate and provide medical care for personnel in the event of a work-related accident or illness; and
- Identify any adverse health effects resulting from hazardous work, and to determine employee fitness for future work assignments;

Based on the examination, the physician identifies any medical restrictions that would affect an employee's ability to safely perform their job. If no restrictions are imposed, the physician certifies the employee as capable of full participation in the work program. The ERT medical surveillance program requires an annual follow-up examination to ensure the continued fitness of field personnel for future work activities. Furthermore, supplemental testing will be administered on an as-needed basis.

3.5 First Aid/ Cardiopulmonary Resuscitation

At least two onsite workers will be currently certified in both Standard First Aid and Adult Cardiopulmonary Resuscitation (CPR) by the American Red Cross or equivalent organization and will include blood borne pathogen training. First-aid training will be updated every 3 years. CPR training will be updated annually.

4.0 GENERAL SAFETY REQUIREMENTS

4.1 SSHP Acknowledgment

All ERT personnel participating in on-site activities at SVAD-050 must read this SSHP and sign the Safety Plan Acceptance form contained in Attachment 1. Photocopies of the signed SSHP Safety Plan Acceptance forms shall be provided to the ERT SSHO by all ERT personnel working on site. The SSHO shall maintain a project file for this documentation.

4.2 On-Site Coordination

Safety meetings shall be conducted at the beginning of field work and on an ad hoc basis thereafter as warranted by site conditions. The purpose of these meetings will be to exchange/update information on the activities planned for the day, anticipated PPE level for each activity, special safety requirements, site conditions, newly identified hazards including temperature, wind velocity and direction, humidity, and precipitation. Records of the daily site meetings will be maintained including attendance and time of the meeting.

4.3 General Safety Rules

The general safety rules listed below apply to all ERT direct personnel, subcontracted personnel and visitors at SVAD-050:

- Eating, drinking, and smoking are prohibited on site;
- All on-site personnel must wear protective clothing appropriate for designated level of protection (i.e., work boots) and personnel shall wash hands before eating and at the completion of work activities;
- An adequately stocked first-aid kit will be maintained; and
- All accidents, injuries, or possible exposures will be reported to the SSHO immediately. A copy of the Accident Report Form is included in Attachment 4.

4.3.1 Buddy System

Most activities at the site will be conducted with a buddy who is able to:

- Provide his or her partner with assistance;
- Observe his or her partner for signs of chemical or heat/cold exposure;
- Periodically check the integrity of his or her partner's PPE; and
- Notify the PM or others if emergency help is needed.

A minimum number of personnel for efficiency and safety will be present in the EZ.

4.3.2 Disciplinary Procedures

Strict adherence to the SSHP will be required for all employees and subcontractors working under ERT's SSHP. Disciplinary action will be enforced for those employees and subcontractors not complying with the project's safety requirements.

5.0 ACTIVITY HAZARD ANALYSIS

The potential hazards associated with the project site include chemical, physical, and biological hazards. The potential for encountering chemical hazards will depend on the types and quantities of chemicals present and the type of work being performed. The potential for encountering physical and biological hazards will depend on the location and type of work being performed. The hazard assessment in this section is intended to communicate to site personnel the chemical, physical, and biological hazards and risks associated with site work. Attachment 3 provides Activity Hazard Analyses for all hazards associated with the activities to be conducted under this project.

5.1 Chemical Hazards

Hazards associated with site activities are varied and include contact with potentially contaminated soil gas/vapor, potentially contaminated creek water and sediment, materials handling, slips and falls, cuts and punctures.

The general contaminants of concern at the SVAD-050 site are:

- VOCs;
- Explosive Compounds;
- Chlorides and Nitrate/Nitrite Compounds; and
- Heavy Metals, including: Lead, Cadmium, and Chromium.

The main routes of exposure for field personnel include:

- Inhalation of contaminant vapors;
- Inhalation of contaminated particulate matter;
- Ingestion of contaminated material; or
- Dermal absorption of contaminated material.

Due to the nature of the contaminants, local features, and type of site activities planned:

- There is a moderate to high potential for inhalation of contaminated particulate matter,
- There is a low to moderate potential for ingestion of contaminated material; and
- There is a moderate potential for dermal contact with contaminated material.

Site personnel can reduce their exposure potential by:

- Using the proper PPE;
- Practicing contamination avoidance;
- Following proper decontamination procedures; and
- Observing good personal hygiene.

5.1.1 Volatile Organic Compounds

VOCs present the greatest exposure potential to field personnel. Due to the high vapor pressure of many of these compounds, inhalation is the primary route of exposure. Skin absorption is also a possible route of exposure, leading to the same symptoms as inhalation overexposure. These compounds may cause central nervous system (CNS), lung, and blood vessel damage.

5.1.2 Explosive Compounds

The explosive compounds that have been identified at SVAD-050 are 2,4-dinitrotoluene (DNT), 2-Amino-4,6-DNT, and 4-Amino-2,6-DNT in very low concentrations. The major concerns with these compounds are flammability and dust dispersion. There are no health concerns for inhalation, skin exposure, or eyes due to the low concentrations and aqueous nature of these contaminants.

5.1.3 Chlorides and Nitrate/Nitrite Compounds

Although these compounds are commonly found in the natural environment, their presence in groundwater above background levels can indicate contamination from a man-made source.

Adverse health effects from chlorides and nitrate/nitrite compounds can result from ingestion of excessive quantities. Chlorides bind with both simple and complex molecules, increasing their health effects. Nitrates are considered nontoxic. However, bacteria in the normal human digestive system can convert nitrates to nitrites. Excessive nitrites in the bloodstream combine with hemoglobin, reducing the blood's capacity to carry oxygen, producing methemoglobinemia.

Due to the low concentrations of chlorides and nitrates and nitrites in the groundwater and the minimal risk for these compounds to be ingested, no health risks are anticipated.

5.1.4 Heavy Metals

High concentrations of heavy metals may cause toxic effects through the inhalation of contaminated dust, or ingestion of contaminated material. General adverse health effects associated with heavy metals overexposure include increased incidence of respiratory cancers, mental disturbances, loss of coordination, respiratory irritation, and metal fume fevers. Heavy metals have the potential for bioaccumulation within the body. The health hazards associated with specific heavy metals include:

- Cadmium – Is a carcinogen (producing lung tumors) and a human poison via inhalation. Inhalation of dust or fumes affects the respiratory tract and kidneys. The dust ignites spontaneously in air.
- Chromium – Dermal contact may cause dermatitis and penetrating skin ulcers. Inhalation of dust may lead to perforation of the nasal septum, and inflammation of the larynx and liver. Hexavalent chromium (Cr^{+6}) is a human carcinogen.
- Lead – Chronic exposure to elemental lead may cause the following symptoms: anemia, dullness, restlessness, irritability, headache, muscular tremors, and loss of memory. Acute exposure may cause permanent CNS damage. It poses a potential health risk through dermal exposure to lead-contaminated waste or soil. Inhalation of dust is also a potential exposure route.

5.2 Hazard Communication

An activity hazard analysis (AHA) will be reviewed with applicable site workers prior to each task. Site-specific activity hazard analyses will be submitted as addendums to this SSHP.

Material Safety Data Sheets (MSDS) for common contaminants will be supplied and kept onsite by the SSHO. Copies of these MSDSs are presented in Attachment 2 of this Plan. Additional MSDSs for materials used during the sampling investigation such as alconox and hydrochloric

acid are also presented. Employees and subcontractors will be informed of the location of MSDSs. Chemicals brought onsite will be properly labeled in accordance with OSHA's Hazard Communication requirements (29 CFR 1926.59), and a MSDS will be provided to the SSHO for each hazardous substance brought onsite.

5.3 Physical and Biological Hazards

Physical hazards, to be discussed in depth in the following sections, will potentially be present at the site during field activities. These physical hazards may include, but not be limited to the following:

- General Physical Hazards
- Fire/Explosion Hazards
- Noise Hazards
- Electrical Hazards
- Utility Hazards
- Weather Hazards
- Cold Stress/Heat Stress
- Material Handling/Moving/Lifting
- Confined Space Hazards
- Biological Hazards

The site will be visually inspected for the presence of general safety hazards (e.g., trip/slip hazards, unstable surfaces or steep grades, sharp objects) prior to beginning work. If hazards are present, these hazards will be recorded and precautionary measures will be taken to prevent injury.

5.3.1 General Physical Hazards

The work site may include many basic physical safety hazards, such as:

- Steep grades and/or uneven terrain, posing slip, trip, and fall hazards
- Holes, ditches, etc., posing fall, cave-in, and other hazards
- Precariously positioned objects, which may cause crushing or other injuries
- Sharp objects (e.g., nails, metal shards, glass), which may cause cuts, injection, or other injuries
- Slippery surfaces, posing slip and fall hazards
- Unstable surfaces (e.g., walls that may cave-in, unstable underground structures) which may pose fall, crushing, or other injuries

ERT personnel will observe and abide by access restrictions, zones, and the safety requirements of this SSHP.

Other hazards such as weather conditions will be evaluated on a daily basis by ERT's SSHO, and appropriate precautions will be identified. There are several site-specific conditions that should be noted by anyone working on the site during the pre-design investigation. Extra care should be taken in these situations to work in a safe manner. These are discussed briefly below.

5.3.2 Fire/Explosion Hazards

Explosion and fire hazards may be present at the work site due to the unexpected ignition of chemicals or fuels, the sudden release of materials under pressure, or due to unauthorized smoking in work areas. Site operations will be conducted in accordance with local fire codes and regulations.

Field vehicles will be equipped with fire extinguishers. Employees will be trained in the proper use of fire suppression equipment. Large fires, not able to be controlled with a fire extinguisher, will be handled by the local fire department. In the event of a fire, employees will contact the fire department, property owner, and SSHO and will report to the pre-determined rally point unless this location has become dangerous due to the existing emergency. Employees shall always be on the alert for unexpected events, and be prepared to act in these emergencies.

5.3.3 Noise Hazards

Work around heavy equipment often creates excessive noise. Noise can cause workers to be startled, annoyed, or distracted; it can cause physical damage to the ear, pain, and temporary and/or permanent hearing loss; and it can interfere with communication. If workers are subjected to noise exceeding an 8-hour time-weighted average (TWA) sound level of 85 decibels on the A-weighted scale (dBA), hearing protection will be provided with an appropriate noise reduction rating to comply with 29 CFR 1910.95 and reduce noise levels to or below 85 dBA. The current scope of work includes vegetation clearance using machinery, MEC clearance and the demolition of identified MEC, soil sampling, and groundwater sampling, therefore, noise hazards are not anticipated. Personnel involved in the detonation of identified MEC will be a sufficient distance from the detonation site so that noise issues will be limited. However, if needed, hearing protection will be selected by the SSHO with an appropriate Noise Reduction Rating (NRR) to reduce noise levels to or below these values.

5.3.4 Electrical Hazards

Overhead power lines, electrical wiring, electrical equipment, and buried cables pose risks to workers of electric shock, burns, muscle twitches, heart fibrillation, and other physical injuries, as well as fire and explosion hazards. In addition, electrical wiring and electrical equipment associated with sampling activities may pose a hazard.

In accordance with OSHA's standard for Electrical Safety Related Work Practices (29 CFR 1910.331-.335), protective measures will be taken when working near live electrical parts, including but not limited to:

- Insulation and guarding of live parts
- Grounding
- De-energizing live parts followed by lockout/tag out to prevent inadvertent re-activation of the parts
- Electric protective devices (e.g., insulated tools)
- Safe work practices, including:
  - Inspection of work area to identify potential spark sources
  - Maintenance of a safe distance from live electrical parts as per USACE requirements:

Minimum Clearance From Energized Overhead Electric Lines

Nominal System Voltage	Minimum Rated Clearance
o 0-50 kilovolt (kV)	o 9.8 ft (3m)
o 51-200 kV	o 14.7 ft (4.5 m)
o 201-300 kV	o 19.7 ft (6m)
o 301-500 kV	o 24.6 ft (7.5 m)
o 501-750 kV	o 34.4 ft (10.5 m)
o 751-1,000 kV	o 44.3 ft (13.5 m)

(Source: USACE, 2003)

- Proper illumination of work areas
- No "blind reaching" around live electrical equipment
- Provision of barriers, shields, or insulation to prevent inadvertent contact with live parts
- Use of nonconductive, intrinsically safe equipment near live parts

Field personnel will not be permitted to work on electric parts of equipment that have not been de-energized, locked out, and tagged by personnel trained to work with electrical equipment. Field personnel will not work near energized exposed live parts without the use of protective measures, nor will need for this to occur be expected during the scheduled field activities for this project.

Lightning may be an electrical hazard during outdoor operations, particularly for workers in open areas and handling metal equipment. Weather conditions will be monitored and work suspended during electrical storms at the discretion of the SSHO and Site Manager.

MEC disposal activities planned for SVAD-050 include electric-firing detonation of items. Electrical hazards associated with the electric-firing methods are summarized in Appendix C (MEC Investigation Plan) of the main Work Plan document.

5.3.5 Utilities

Underground utilities are not anticipated in the areas being investigated in the RI. Illinois One-Call will be notified to ensure utilities do not exist. However, field personnel will be aware of unanticipated hazards. ERT reserves the right to conduct further utility clearance procedures as necessary.

5.3.6 Weather Hazards

Weather conditions will be taken into consideration during site activities. Heavy rains, snowfall, freezing conditions, electrical storms, high winds, and extreme temperatures may create dangerous situations for employees. Inclement weather may also impair equipment performance. Whenever unfavorable conditions arise, the Site Manager and SSHO will evaluate both the safety hazards and the ability of the employees to effectively perform given tasks under such conditions. Activities will be halted at the discretion of the SSHO and Site Manager. Site activities will cease when lightning has been sighted in an approaching storm.

Wind direction will be considered when positioning for sampling and demolition efforts associated with located MEC. If exposure to contaminated emissions is anticipated, workers will locate upwind. Wind direction often changes abruptly and without warning, so personnel will be prepared to reposition, if necessary.

5.3.7 Cold Stress

Cold stress hazards are most likely to occur at low temperatures or low wind chill factors, with wet, windy conditions contributing to the risk. Site workers will respect the OSHA Cold Stress Equation while working in cold conditions. Table 5-1 will be used to determine the degree of cold stress hazard.

Table 5-1. TLVs Work/Warm-up Schedule for Outside Workers based on a Four-Hour Shift*

Air Temperature - Sunny Sky		No Noticeable Wind		5 mph Wind		10 mph Wind		15 mph Wind		20 mph Wind	
°C (approx)	°F (approx)	Max. work Period	No. of Breaks**	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks
-26° to -28°	-15° to -19°	(Norm breaks) 1		(Norm breaks) 1		75 min.	2	55 min.	3	40 min.	4
-29°to -31°	-20°to -24°	(Norm breaks) 1		75 min.	2	55 min.	3	40 min.	4	30 min.	5
-32° to -34°	-25°to -29°	75 min.	2	55 min.	3	40 min.	4	30 min.	5	Non-emergency work should cease	
-35° to -37°	-30° to -34°	55 min.	3	40 min.	4	30 min.	5	Non-emergency work should cease			
-38° to -39°	-35° to -39°	40 min.	4	30 min.	5	Non-emergency work should cease					
-40° to -42°	-40°to -44°	30 min.	5	Non-emergency work should cease							
-43° & below	-45° & below	Non-emergency work should cease									

**2008 TLVs and BEIs - Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices. Cincinnati: American Conference of Governmental Industrial Hygienists (ACGIH), 2008 - page 213*

*** The schedule assumes that "normal breaks" are taken once every two hours. At the end of a 4-hour period, an extended break (e.g. lunch break) in a warm location is recommended.*

Workers will be familiar with the signs and symptoms of cold stress, which include:

Hypothermia - Cold-induced decreasing of the core body temperature that produces shivering, numbness, drowsiness, and muscular weakness. If severe enough, it can lead to unconsciousness and death.

Frostbite - Constriction of blood vessels in the extremities, decreasing the supply of warming blood. This may result in formation of ice crystals in the tissues, causing tissue damage. Conditions may range from frostbit, which is a numbing of extremities, to deep-freezing tissue beneath the skin. Symptoms include white or grayish skin, blisters, numbness, mental confusion, failing eyesight, fainting, shock, and cessation of breathing. Death may occur from heart failure.

Pain in the extremities may be the first warning of cold stress, and precautions will be taken to reduce exposure. Severe shivering will be taken as a sign of immediate danger to the worker, and exposure to cold will be immediately terminated. Personnel exhibiting signs and symptoms of cold stress will be removed from the site, decontaminated, and given appropriate first aid. Emergency medical services will be contacted if symptoms are severe (e.g., more than numbness of the extremities or shivering). When air temperatures are less than 36ºF (including wind chill), workers who become immersed in water or whose clothing becomes wet will be immediately provided a change of clothing and be treated for hypothermia.

As a precautionary measure, employees will wear layers of loose-fitting clothing including insulated coveralls, head cover (perhaps a wool cap beneath a hard hat), and boots when temperatures fall below 40°F, including wind chill. Head covering for use beneath a hard hat must be specifically designed for that purpose so that the function of the hard hat is not impaired. Protection of the hands, feet, and head is particularly important because these are likely to be injured first by cold. However, actual injury to hands, feet, and head is not likely to occur without prior development of early signs of hypothermia such as numbing and shivering. Bare skin contact with cold surfaces (below 32°F) will be avoided. Personnel will wear wind-resistant outer shell to decrease wind chill effects. Less than one minute of exposure to cold is permitted when the air speed and temperature results in an equivalent chill temperature of -26ºF to -72ºF.

A temperature-dependant work regimen limiting lengthy periods of outdoor activity may be necessary. Workers entering heated shelters will remove the outer layer of clothing and loosen remaining clothing to permit the evaporation of perspiration. Workers will avoid dehydration by drinking water or other decaffeinated beverages, including warm drinks and soups.

5.3.8 Heat Stress

Heat stress hazards can occur even in temperatures not commonly considered ‘hot” due to the level of physical activity, the level of PPE the worker is wearing, or the physical condition of the worker. Illness resulting from exposure to extreme heat is possible during field operations. Factors affecting heat stress include high temperatures and humidity, direct sun or heat, limited air movement, physical exertion and poor physical condition. Personnel, especially those in impermeable clothing (i.e., chemical suits), will be familiar with the signs and symptoms of heat stress, including:

- **Heat Cramps** - Muscle spasms in the abdomen or limbs. Frequent rest periods and fluid intake are appropriate measures to prevent or reduce heat cramps.
- **Heat exhaustion** - Severe dehydration; pale, clammy skin; profuse sweating; dizziness, light-headedness; slurred speech; rapid pulse; confusion; fainting; fatigue; cool skin; nausea. Affected personnel will be escorted from the site, decontaminated promptly, set in cool, shaded area, and given fluids slowly.

- **Heat stroke** - Life-threatening condition occurring when the body's temperature-regulating system improperly functions. Hot dry skin; rapid, deep breathing; lack of perspiration; delirium; high fever (often 106ºF or more), nausea; unconsciousness. Brain damage and/or death may occur, if body temperature is not reduced. Provide fluids, use cooling devices (hose-down or shower), call emergency medical services or transport to hospital immediately.

Some preventive measures to avoid heat stress include:

- Frequent resting in cool or shaded areas
- Prevent heat stress by resting frequently in a shaded area and consuming large quantities of fresh potable water (more than amount needed to simply quench thirst). Drink at least 8 ounce (oz) of water or diluted Gatorade every 2 hours when temperatures exceed 75ºF. – do not consume alcoholic beverages to combat dehydration or heat stress.

Heat stress monitoring will be conducted in a manner that anticipates and prevents the onset of heat stress symptoms (i.e., wet-bulb globe temperature (WBGT) monitoring coupled with OSHA work-rest regimens). If the WBGT Index is used and semi-impermeable or impermeable clothing is worn, the WBGT Index will be lowered, as appropriate.

Non-acclimated workers and workers wearing full body impermeable chemical protective clothing shall be monitored when the work area temperature is greater than 70°F. The worker's heart rate and blood pressure will be measured at the start of a rest break, and the work period will be decreased so that after 1 minute of rest, a worker's heart rate (HR) does not exceed 110 beats per minute. Heart rate will be monitored using a stethoscope and sphygmomanometer.

A suggested work-rest regimen is:

Ambient Temperature	Work	Rest
70˚F	3 hours	15 min
75˚F	2-1/2 hours	15 min
80˚F	2 hours	15 min
85˚F	1-1/2 hours	15 min
90˚F	1 hour	15 min
95˚F	½ hour	15 min

If a worker's HR is greater than 110 beats per minute, the next work period will be shortened by 33 percent, while the length of the rest period stays the same. If the HR is 110 beats per minute at the beginning of the next rest period, the following work cycle will be shortened by 33 percent. When ambient temperatures are expected to exceed 75°F, the resting HR of each worker will be measured prior to the start of onsite activities.

Other factors, such as a worker's level of acclimation, level of physical fitness, and age, may increase or decrease his susceptibility to heat stress. Before assigning a task to an individual worker, these factors will be taken into account to ensure that the task will not endanger the worker's health.

If a heat-related illness is suspected or observed, the affected person will be moved to a cool or shaded area and given plenty of liquids to consume. If symptoms of a heat stroke are observed, the victim will be cooled and immediately and call 911.

5.3.9 Material Handling/Moving/Lifting

Materials handling at the subject site could vary from heavy equipment handling to manually moving/lifting items. Hazards associated with materials handling include physical injury, detonation, fire, explosion, and vapor generation among others. During the sampling activities, it is anticipated that personnel will be moving sample equipment (i.e., pumps) and coolers.

Injuries to back and abdominal muscles from improperly lifting of loads are the most common occupational injuries reported. Such injuries can range from relatively mild strains to major, permanently disabling injuries. Before lifting a load, personnel will consider the overall weight, distribution of weight, awkwardness of the load, distance to be carried, obstacles to be negotiated, site conditions, and visibility.

When using equipment to move objects or materials, the proper work practices will be followed. Equipment used will be designed for the task to be performed. Equipment will be inspected regularly by the SSHO and the Site Manager, and damaged or defective equipment will be removed for service. Planning is critical when handling materials. The Site Manager, in conjunction with the Project Manager, will plan where the materials are to be moved, taking into consideration the current location of such materials and hazards associated with moving them. Routes for moving materials will be clearly communicated, with paths cleaned of obstructions so materials may be transported.

Loads will be lifted using the power of the leg muscles rather than the back, stomach, or arm muscles. The item will be approached to balance the load evenly. Backs will be kept straight and the arms nearly parallel with the body. The knees will be bent to grasp the load. Lifting will be done by straightening the legs without bending the body, holding the load as close to the body as possible and the back remaining as straight as possible. When possible, a lifting arm or automatic lift gate will be used to move drums into and out of trucks.

Bulky, heavy loads will be handled by at least two people, ensuring that the load is level and evenly distributed between personnel helping to carry it. Carriers will know the destination and path for the load.

5.3.10 Slips, Trips, and Falls

Working in and around the site will pose slip, trip and fall hazards due to possibly slippery conditions. The majority of RI activities will be conducted in areas of known debris and overgrown vegetation. Extreme caution should be used when traversing and working in such conditions. Special precautions should be taken to prevent slips, trips and falls.

To prevent slips and falls, a high coefficient of friction between the shoe and walking surface is required. On icy, wet, and oily surfaces, the coefficient of friction is extremely low with shoes that are not slip resistant. A coefficient of friction of 0.40 to 0.50 or more is needed for excellent traction. As reference, a brushed concrete surface and a rubber heel will often show a coefficient

of friction greater than 1.0. Leather soles on a wet smooth surface, such as ceramic tile or ice, may have a coefficient of friction as low as 0.10.

Providing dry walking and working surfaces and slip-resistant footwear are the answer to slips and their resultant falls and injuries. Obviously, high heels, with minimal heel-to-surface contact, taps on heels, and shoes with leather or other hard, smooth-surfaced soles lead to slips, falls, and injuries. Shoes with rubber-cleated, soft soles and heels provide a high COF and are recommended for most agricultural work.

In addition to wearing the wrong footwear, there are specific behaviors which can lead to slips, trips, and falls. Walking too fast or running can cause major problems. In normal walking, the most force is exerted when the heel strikes the ground, but in fast walking or running, one lands harder on the heel of the front foot and pushes harder off the sole of the rear foot

Other problems that can lead to slips, trips and falls are: distractions; not watching where one is going; carrying materials which obstruct view; wearing sunglasses in low-light areas; and failure to use handrails. These and other behaviors, caused by lack of knowledge, impatience, or bad habits developed from past experiences, can lead to falls, injuries, or even death.

5.4 Biological Hazards

There may be a possible hazard arising from poisonous plants, such as poison ivy, and from some animals, such as snakes, rats, and insects such as ticks. ERT personnel should avoid all contact with animals.

All ERT personnel will be trained to identify poison ivy during the preliminary site safety meetings.

Insects, including bees, wasps, hornets, spiders, and ticks may be present at the Site making the chance of a bite possible. Some individuals may have a severe allergic reaction to an insect bite or sting that can result in a life threatening condition. Personnel that have been bitten or stung by an insect at the Site should notify the ERT SSHO of such immediately. The following is a list of preventive measures:

- Apply insect repellent prior to fieldwork and as often as needed throughout the shift.
- Wear proper protective clothing (work boots, socks and light colored pants).
- When walking in wooded areas, to the extent possible avoid contact with bushes, tall grass, or brush.
- Field personnel who may have insect allergies (i.e., bee stings) should provide this information to the ERT SSHO or his designee prior to commencing work, and shall have allergy medication on Site.

The ERT SSHO will instruct the project personnel in the recognition and procedures for encountering potentially hazardous insects at the Site.

5.4.1 Poisonous Plants

Poisonous plants, such as poison ivy, poison oak, or poison sumac may be present at various work sites. Personnel are expected to know how to recognize these plants and avoid them. If

these plants need to be removed to obtain a work area free of hazards, gloves will be worn to protect against the poisonous oils of the plant, and the plant will be placed in a trash bag and disposed of in a proper receptacle. Never burn a poisonous plant; smoke and vapors are also poisonous when inhaled. If dermal contact occurs, the affected areas will be washed with soap and water immediately.

5.4.2 Insect Bites/Stings

Protective outer clothing such as gloves, hard hats, and coveralls can reduce the potential for insect bites and stings. Avoid disturbing insect nests encountered on the job site if possible, as an insect swarm may develop. If an insect nest (i.e., a bee hive) is encountered in an area that is likely to be disturbed during site activities, the nest will be eradicated prior to site activities to ensure employee safety.

Insect bite symptoms may include redness, rash, swelling, chills, fever, diarrhea, and vomiting. A worker who has been bitten or stung and shows symptoms of a severe reaction will seek medical assistance immediately. Workers with allergies to insects (i.e. bees) should advise the SSHO prior to field activities and should carry an antidote kit, if necessary.

To prevent contact with fleas or disease-carrying ticks (deer ticks), workers should wear long-sleeved shirts, long pants, and boots that extend above the ankle with pants cuffs taped to boots, especially when working in wooded, overgrown, or high grass areas. Workers will thoroughly check clothing, skin, and hair for the presence of ticks at the end of each workday. If a tick attaches to the body, it will be removed by gently tugging with tweezers where the mouth-parts enter the skin. The tick should not be killed prior to removal. Ticks can carry a variety of diseases, such as Lyme, Rocky Mountain spotted fever, ehrlichiosis, and babesiosis. If a worker suspects he has been bitten by a tick, the area shall be monitored for several days/weeks by the individual for the development of a noticeable bulls-eye shaped skin rash at the affected area. If this develops, seek immediate medical attention for a possible infection of Lyme disease. Insect repellant containing *DEET®* is an effective means of tick control.

Mosquitoes infected with the West Nile Virus have been identified throughout Illinois. Field personnel will acquaint themselves with the symptoms associated with West-Nile Virus and will contact a physician, as well as the ERT SSHO, if the disease is suspected.

Lyme disease is caused by infection from a deer tick that carries a spirochete. During the painless tick bite, the spirochete may be transmitted into the bloodstream, which could lead to the worker contracting Lyme disease. This flu like illness commonly happens between May and October when ticks are more active. Symptoms can include a stiff neck, chills, fever, sore throat, headache, fatigue and joint pain. Early signs may include an expanding skin rash and joint pain. If left untreated, Lyme disease can cause serious nerve or heart problems as well as a disabling type of arthritis. If personnel feel sick or have signs similar to those above, they should notify the SSHO immediately.

It is recommended that personnel check themselves when in areas that could harbor deer ticks, wear light color clothing and visually check themselves and their buddy when coming from wooded or vegetation covered areas. If a tick is found biting an individual, the SSHO should be

contacted immediately. The tick can be removed by pulling gently at the head with tweezers. The affected area should then be disinfected with an antiseptic wipe.

5.4.3 Animal Bites

Rodents, snakes, stray dogs, stray cats, raccoons or other animals may be encountered on the job site. The only effective measure to preclude animal bites is avoidance. Contact with wild animals will be avoided. Do not reach into an object such as a pipe that may contain a rodent or other animal or attempt to remove the animal. If possible, delay activity in this area until the animal leaves or has been removed by an animal control unit. If it is necessary to use or move an object where an animal is hiding, the SSHO may have to notify the local animal control agency to subdue an animal that may cause a risk to workers (i.e., a raccoon). Do not approach an animal, as this may cause the animal to be in an aggressive state. Persons bitten by an animal will seek medical assistance immediately, especially if it is suspected that the animal may be rabid. Aggressive or disoriented behavior, as well as foaming at the mouth, can be signs of rabid animals. Until medical assistance can be reached, persons will watch for symptoms of severe swelling, nausea, and shock. The local animal control agency will be contacted if a stray dog is observed on the property, especially if its presence will obscure work activity or the animal's safety. Do not attempt to leash a stray dog (no matter how harmless it may appear); doing so would greatly increase the risk of an animal bite.

5.4.4 Bacteria

Adherence to personal protective equipment requirements, personnel decontamination procedures, personal hygiene measures and work zone rules will minimize exposures. Bacteria are not expected to be a hazard based on the location of field activities. However, syringes, medical or biohazard waste encountered in an uncontrolled setting will be reported immediately to the SSHO.

5.4.5 Humans

The site is a gated, remote area and there is limited possibility that non-study team personnel may be encountered in the vicinity of the work site. While unauthorized intruders are not expected to purposely or maliciously pose a threat to personnel working at the site, precautions will be taken to prohibit the presence of unauthorized personnel in the work zone. Onlookers will be asked (repeatedly, if necessary) to maintain a 200-ft distance from the work zone and will be asked to leave the area when such encounters threaten non-compliance with this SSHP. Obstinate or malicious behavior in this regard will be dealt with by calling the police (911). In addition, the USACE Project Technical Manager and ERT Project Manager will be notified.

5.5 Task-Specific Hazards

Tasks associated with the project present unique hazards other than those discussed in previous sections of the SSHP. These task specific-hazards are described in detail in the following subsections.

5.5.1 MEC Clearance

In order to safely access potential geophysical investigation and environmental sampling areas, necessary to complete RI activities, MEC clearance related to previous Department of Defense (DoD) activities is necessary. The clearance of potentially unexploded munitions and explosives is inherently dangerous. Health and safety hazards specifically related to this activity are

described in detail in this section and should be reviewed in detail with the approved MEC Clearance Work Plan.

The greatest hazard with MEC is an unplanned detonation. There is no "safe" procedure for dealing with MEC, merely procedures that are considered less dangerous. Safety in MEC operations will be achieved through strict adherence to applicable safety regulations and quality supervision. ERT's procedures for MEC-related activities will be provided in the MEC Investigation Plan (Appendix C to the RI/FS Work Plan). In addition to complying with the preceding requirements, the regulations and guidance of EP 385-1-95a (USACE, 2004b) will be followed.

5.5.1.1 General Precautions

Tasks that may be performed under this plan will be carried out by UXO qualified personnel and non-UXO qualified personnel. Non-UXO qualified personnel will always be escorted by UXO qualified personnel when working at a site when there is potential for UXO/MEC to be present at the work site. Non-UXO qualified personnel will never handle a UXO item and will always follow safety precautions listed below. UXO qualified personnel will only handle those MEC items that have been deemed safe to move and will always follow safety precautions that are listed below. There are three general principles or precautions to mitigate hazards associated with MEC:

- The cardinal principle or precaution to be observed involving explosives, ammunition, or severe fire hazards is to limit the exposure to a minimum number of personnel, for the minimum amount of time, to a minimum amount of hazardous material consistent with a safe and efficient operation.
- The age or condition of an MEC item may not decrease its effectiveness. MEC exposed to the elements for extended periods of time may become more sensitive to shock, movement, and friction because the stabilizing agent may be degraded.
- MEC that has been exposed to fire should be considered extremely hazardous. Chemical and physical changes may have occurred to the contents, rendering it more sensitive to detonation than in its original state.

5.5.1.2 On-Site MEC Instructions for General Site Workers without UXO Support Personnel

In general, general site workers will not enter areas with a known or suspected MEC presence without UXO support personnel present or in areas in which the access routes and work areas have not been cleared and clearly marked by UXO support personnel. When MEC material is found or suspected in areas not suspected of containing an MEC presence and UXO support personnel are not physically present, perform the following:

- Immediately stop at the location in which the MEC item or suspect item is observed.
- Do not touch or disturb the MEC item or suspect item in any fashion.
- Do not use cell phones or radios in the discovery area.
- Attempt to mark the general discovery area without placing anything in direct contact with a suspect item.

- Immediately depart the area the same way you entered being aware of your surroundings (Walk, Don't Run!).
- Report the discovery to a UXO qualified person.
- Do not operate vehicles outside of boundaries or roadways that have not been previously cleared by UXO technicians.

5.5.1.3 General Site Workers in Known or Suspected UXO Areas under Qualified UXO Personnel Supervision

- Closely follow instructions provided by UXO qualified personnel.
- Do not pick up, kick, step on, come in proximity to, or in any fashion disturb any foreign object without the express permission of a UXO qualified technician.
- Do not transmit by radio or cellular phone without the express permission of a qualified UXO technician.
- Do not stray beyond the boundaries of previously marked/cleared areas or leave the immediate vicinity of a lead UXO escort.
- Do not permit any object to penetrate the ground unless the penetration location has been surveyed and cleared by a qualified UXO technician.

5.5.1.4 On-Site General MEC Instruction for UXO Personnel

When MEC is discovered and identified, observe these general precautions:

- DO NOT rely on color code for positive identification of known or suspected MEC items or their contents.
- DO NOT touch or move any unknown MEC item regardless of its marking or apparent condition.
- Always assume MEC items contain a live charge until it can be determined otherwise.
- DO NOT continue to work at a known or suspected MEC site if an electrical storm is occurring or approaching. If a storm approaches, leave the site immediately and seek shelter.
- DO NOT walk across a known or suspected MEC site where the ground cannot be seen. If dead vegetation or animals are observed, leave the area immediately.
- Use clearly marked driving lanes, DO NOT drive vehicles into a known or suspected MEC site.
- Attempt to positively identify the MEC item. Take photographs and make notes of all markings or any other identifiers.
- DO NOT be misled by markings on the MEC item stating "practice bomb, "dummy" or "inert." Even practice bombs have a lethal explosive charge used to mark or spot the point of impact.
- DO NOT jar any MEC item, regardless of its apparent condition.
- Report all MEC discoveries to the USACE MEC Safety Specialist.
- DO NOT move an MEC item without the express approval of the USACE MEC Safety Specialist.

5.5.2 Brush Removal

In order to perform activities associated with the RI in a safe manner, brush clearance will be conducted in areas of SVAD-050. Brush and other obstacles will be addressed if they pose a hazard to site workers. All personnel working with tools either mechanical or otherwise will be trained in the proper and safe operation of the equipment. Proper and safe operation will be defined as operation in accordance with the manufacture's recommendation and in accordance with the USACE EM 385-1-1 (2003). Appropriate level D PPE will be required at all times during brush clearance.

Rotary drum and disc-type brush chippers will provide sufficient safety features including sufficient height of feed chute side members to prevent operator contact with the blades, quick stop mechanism and reversing device on the in-feed and anti-kickback mechanism. Brush chipper cutting blades will be kept sharp and otherwise maintained in accordance with the manufacture's recommendations

Power saws will be operated in accordance with the manufacturer's recommendations. The engine of the saw will not be started until all co-workers are clear of the saw and aware of the pending action. The saw will promptly turned off once cutting is complete. The saw will be turned off when not in use or when transporting the saw. Idle speed is not considered off.

All chopping tools will be used in accordance with the manufacture's recommendations. Chopping tools will be inspected regularly for defects such as loose or cracked heads and shafts. When operating a chopping tool, it should always be swung away from the feet, legs, and body.

5.5.3 Drilling and Well Installation

There are inherent hazards associated with the drilling and installation of groundwater monitoring wells (MWs).

- Overhead Hazards: There will be overhead hazards associated with the drilling and installation of the permanent MWs. Appropriate Level D PPE such as a hard hat will be worn at all times while on site.
- Noise hazards will be present which are associated with the drilling and installation of the MW. It is likely that workers will be subjected to noise exceeding an 8-hour TWA sound level of 85 dBA, therefore, hearing protection will be worn at all times while the drill rig is in operation with an appropriate noise reduction rating to comply with 29 CFR 1910.95 to reduce noise levels to or below 85 dBA. Hearing protection will be selected by the SSHO with an appropriate Noise Reduction Rating (NRR) to reduce noise levels to or below these values. Appropriate hearing protection will be worn at all times while heavy equipment (e.g., drill rig) is in use in accordance with 29 CFR 1910 and 29 CFR 1926.
- As there will be drilling activities associated with this work, there is the potential for underground/electrical hazards. However, Illinois Miss Utility will be contacted to mark-out public utilities. In addition, ERT reserves the right to use ground penetrating radar (GPR) equipment (GSSI SIR-3000) in those areas in an effort to identify underground utilities and obstructions. Should any underground/electrical hazards be encountered during drilling activities, the Site Manager and/or the SSHO will immediately stop the work and contact the Project Manager and a decision will be made as to the most appropriate way to proceed.

- Depending on the particular drill rig employed, drilling operations can present exposure to flying objects and dust. Measures used to control such exposures will include the use of water or other misting apparatus to keep dust down, or the use of a guard installed around the drill to protect against flying objects and dust.
- Assembly and disassembly of rigs, rotary and auger drilling, and grouting apparatus can cause potential hazards to exposed appendages and unprotected body parts. Extreme caution is to be observed and undisturbed focus maintained while these activities are being performed. All appropriate tools and methodology will be used to conduct assembly and disassembly. No equipment shall be used that is not properly secured and/or assembled. Any indication that the equipment has not been assembled and/or disassembled properly should be expressed to the team leader and activities stopped until all issues are resolved.
- During the operation of drilling equipment, soil, wood fragments and other artifacts can potentially get caught in the flights of the augers. Under no circumstance should these object(s) be cleared while the drill rig is in operation. If the augers are to remain fastened to the drill rig while the artifacts are removed, it will be done so using a pressure washer, brush or other device to avoid hand contact with the auger.
- During the heavy soils removal of the decontamination process, if the auger is to be decontaminated while remaining attached to the drill rig, it will be done so using a pressure washer, brush or other means to avoid hand contact with the auger. The preferred method of decontamination, when applicable, is to remove the augers from the drill rig, mechanically remove all artifacts from the flights of the auger and then proceed with the specified decontamination process.

6.0 SITE ACCESS

6.1 Site Access

Site access will be coordinated with the USACE Project Engineer.

6.2 Site Control

During the field investigation, the following activities will be performed:

- Site reconnaissance.
- Surveying and vegetation clearing
- MEC clearance
- Geophysical investigation
- Well installation, purging and groundwater sampling.
- Soil, sediment and surface water sampling.

Areas associated with the MEC disposal will be closed to all personnel not essential to the task being conducted in that area. UXO-qualified personnel will be considered essential personnel during intrusive activities. Environmental sampling and geophysical investigation personnel will be considered nonessential personnel and their activities will be coordinated with the EZ requirements. All personnel engaged in site activities will attend a daily safety briefing given by the ERT UXOSO. All ERT and subcontracted personnel will receive site-specific training prior to starting work on the site. Unescorted access will be restricted to several areas of the site. Access to areas being intrusively sampled for MEC will be restricted. Only personnel essential to the intrusive sampling process will be allowed inside the EZ. Environmental sampling personnel will be escorted by UXO Technicians. Intrusive operations will be stopped if nonessential personnel or non-UXO qualified personnel are inside the EZ.

Area closures will be coordinated and approved by the USACE and SVDA officials. Barricades will be removed immediately after completion of UXO operations. Caution tape that is not considered usable will be disposed of through normal trash disposal.

6.2.1 Activities Not Requiring Work Zones

Activities not requiring specific work zones include:

- Site reconnaissance.
- Geophysical investigation

6.2.2 Activities Requiring Work Zones

Well purging and sampling, soil sampling, MEC investigation and disposal will require the establishment of work zones. The three separate work zones that will be established for each of these activities include:

- The exclusion zone.
- The contamination reduction zone.
- The support zone.

The exclusion zone (EZ) for well purging and sampling will consist of a 10-ft radius from the well. EZs will also be verbally explained and maintained to site visitors or onlookers. Proper PPE will be worn when working in the EZ (see Section 6.0).

The contamination reduction zone shall be immediately adjacent to the exclusion zone and shall have equipment for appropriate decontamination and receptacles for used disposable supplies. Based on previous investigations, the expected concentrations of contaminants suggest personnel decontamination will consist of simply removing and disposing of PPE or removal and bagging of soiled outer garments (e.g., coveralls), if worn. Potable water and an eye wash solution will be available in the contamination reduction zone.

If elevated levels of contaminants are encountered, as discussed in Section 6, work will stop and the site will be evaluated. If protection is to be upgraded, the exclusion zones and contamination reduction zones shall become more stringent. The exclusion zone will be located within 25 ft of field-investigation activities where respiratory protection is required. The contamination reduction zone will be expanded to include a non-phosphate detergent scrub station and potable water rinse station prior to removing and disposing of chemically protective coveralls, boot covers, and outer gloves.

The support zone shall consist of the site vehicle and will also contain emergency response equipment including phone and first aid supplies.

7.0 SAFETY MEETINGS

Employees, subcontractors, and site visitors will read this SSHP and will indicate their understanding of the requirements by signing the SSHP Review Record which is contained in Attachment 1. The SSHO will brief employees on the potential hazards at the site and protective measures to be implemented, both prior to entry and daily during the work. An evacuation rally point will be used in the event of an emergency and will be communicated to personnel. This location will be an upwind point from site activities, in an area not expected to be affected by an emergency situation onsite and will be determined in the field by the SSHO. The SSHO will brief visitors of the rally areas prior to initial entry.

7.1 Pre-Entry Briefing / Daily Safety Meeting

The Site Manager and SSHO will provide site-specific training on the contents of this SSHP, including site monitoring equipment; emergency procedures; areas of restricted access; methods of decontamination; responsibilities for safety of personnel, residents, and property; physical, chemical and biological hazards; PPE; and the location of the MSDSs.

7.2 Daily Safety Meetings

Daily safety meetings will be held prior to personnel working on site. Following review of this document and receipt of the pre-entry briefing, personnel shall indicate their understanding of the site-specific hazards and appropriate emergency response by signing the plan review record (Attachment 1).

The SSHO is responsible for conducting safety meetings, daily during field activities, to discuss status of site health and safety. These meetings shall also address employees' concerns regarding onsite safety and hazard control practices and procedures.

8.0 PERSONAL PROTECTIVE EQUIPMENT AND ENVIRONMENTAL MONITORING PROGRAM

The PPE specified herein represents the hazard analysis and PPE selection required by 29 CFR 1910.132. For the purposes of PPE selection, the ERT SSHO is considered qualified to make these decisions. The signatures on the front of the SSHP constitute certification of the hazard assessment.

It is anticipated that only Level D PPE will be required at the Site. Level D protection is applicable when no respiratory protection and minimal skin protection is required. Level D can be used in the following circumstances:

- The atmosphere contains no known hazard
- Work functions preclude splashes, immersion, or the potential for unexpected inhalation of or contact with hazardous levels of any chemicals.

The Level D Recommended Equipment for this site includes:

- Coveralls
- Safety boots/shoes
- Safety glasses or chemical splash goggles
- Hard hat

Optional equipment for Level D includes:

- Gloves
- Escape bottle
- Face shield

8.1 General PPE Requirements

Personnel will be equipped with proper PPE for the tasks they are asked to perform. The SSHO will distribute PPE and will also train employees in proper use and disposal of PPE.

8.2 Initial Requirements / Upgrade or Downgrade of PPE Levels

Based on evaluation of the potential safety and health hazards, the required initial levels of PPE is Level D. The following field activities will require Level D PPE:

- Site recon.
- Well purging and sampling.
- Soil gas sampling.
- Surface water sampling.
- Vegetation clearing
- MEC clearance
- Geophysical investigations

Level D protection will be the minimum protection used for all field operations. The following are the minimum requirements for Level D:

- Steel-toe, steel-shank safety shoes/boots

- Hard hat that meets American National Standards Institute (ANSI) Standard Z89.1-1986 (for use during sampling activities if overhead hazards exist)
- Chemical-resistant gloves when conducting groundwater sampling.
- Leather work gloves (as appropriate)
- Safety glasses
- Hearing protectors (when noise hazards are present or as required by SSHO)
- Face shield (when operating steam cleaner or decontaminating equipment using nitric acid)
- Coveralls (as appropriate)
- Poly-coated coveralls (when splash hazards are present or contact with heavily contaminated water is expected)

If an upgrade in PPE is required, Level C protection is the anticipated upgrade level of protection. The following are the requirements for Level C:

- Full-face or half-mask, air purifying respirators with organic cartridges [National Institute for Occupational Safety and Health (NIOSH) approved].
- Hooded chemical-resistant clothing (overalls; two-piece chemical-splash suit; disposable chemical-resistant overalls).
- Safety glasses
- Coveralls (optional as applicable).
- Gloves, outer, chemical-resistant.
- Gloves, inner, chemical-resistant.
- Boots (outer), chemical-resistant steel toe and shank.
- Boot-covers, outer, chemical resistant (disposable) (optional as applicable).
- Hard hat that meets ANSI Standard Z89.1-1986 (for use during sampling activities if overhead hazards exist).
- Escape mask (optional as applicable).
- Face shield (when operating steam cleaner or decontaminating equipment using nitric acid).

Work will stop if action level(s) are exceeded prior to upgrading to Level C, and the USACE Project Technical Manager and ERT's Project Manager will be notified of the situation.

8.3 Real-Time Monitoring

Organic Vapor Meter (OVM): Real-time monitoring for VOCs will be conducted using an OVM equipped with a photoionization detector (PID). The OVM will be used to monitor employee breathing zones during all invasive activities, including (but not limited to) soil, sediment, and groundwater sampling.

Monitoring the employee's breathing zone will be conducted at least every 15 minutes until the SSHO determines a more applicable schedule. The OVM will also be used to monitor local and area VOC readings during invasive work activities, and prior to entry and periodically during any work within a trench/confined space. VOCs will be monitored using a Thermo-

Environmental Instruments, Inc. Model 580S OVM, or its equivalent, with a 11.2 eV lamp. The 580S OVM has a detection limit of 0.1 ppm as isobutylene.

Combustible Gas Indicator (CGI)/Oxygen Level Meter: Real-time monitoring for combustible gases and oxygen levels may be conducted using a CGI. The CGI will test for the presence of combustible gases by monitoring the lower explosive limit (LEL) of organic vapors.

Real-time monitoring may be conducted prior to, and during, the following types of work activities:

- Sampling of monitoring wells (at the borehole)
- Sampling of surface water.

Sound Level Meter (SLM): At the discretion of the SSHO, an SLM will be used to monitor employee noise levels during heavy equipment and power tool operation, and during other potentially noisy site activities. Hearing protection will be required for employees exposed to 85 dBA. A Metrosonics dB-307 SLM, or its equivalent, will be used on the A-scale/slow response, to monitor for noise levels.

Calibration of Real-Time Monitoring Equipment: Monitoring and calibration protocols will be performed in accordance with the manufacturer's guidelines. Calibration will be performed, at a minimum, prior to each day's use. A copy of each instrument's manual will be kept in the SZ. The following calibration methods will be used:

- The OVM will be calibrated using either a 100 ppm or a 250 ppm isobutylene span gas concentration.
- The CGI/Oxygen Level Meter will be calibrated using a gas mixture which represents 50 percent (50%) LEL and 15 percent (15%) oxygen.
- The SLM will be calibrated using an acoustical calibrator with a known decibel level output.

8.4 Action Levels

Tables 8-1 through 8-3 list the action levels and action requirements for the OVM, CGI, and the SLM. Upgrading respiratory protection is based on maintaining the upper limit of the action level for approximately 1 to 5 minutes (i.e., a non-transient reading), or at the discretion of the SSHO.

Table 8-1. OVM Action Levels	
Meter Response	**Action**
OVM response < 5 ppm above background	No respiratory protection required (i.e., Level D)
OVM response > 5 ppm and < 25 ppm above background	Upgrade to Level C respiratory protection
OVM response > 25 ppm above background	Retreat from work area*

Table 8-2. CGI/Oxygen Level Meter Action Levels	
Meter Response	**Action**
CGI response <10% LEL	Continue normal operations
CGI response >10% and <20% LEL	Eliminate all sources of ignition from work area; implement continuous monitoring
CGI response >20% LEL	Discontinue operations; allow to vent
Oxygen Level Meter response < 19.5 % or > 23.5 %	Retreat from work area*

*Note: If a retreat becomes necessary, the CRSD HSM will be consulted in regard to upgrading to Level B respiratory protection, adding mechanical ventilation, or possible changes in work practices.

Table 8-3. SLM Action Levels	
Meter Response	**Action**
SLM response <85 dBA	Proceed with periodic monitoring
SLM response >85 dBA	Hearing protection is required

During field operations, the SSHO will evaluate environmental monitoring results and potential for exposure to hazards. The SSHO can authorize an upgrade or downgrade of the PPE level worn onsite. Changes in PPE level, including the rationale for the changes, shall be documented by the SSHO. If PPE is upgraded to Level C, respirator cartridges will be changed at least daily or more frequently if damaged, soiled, or if breathing resistance increases.

8.5 Inspection and Maintenance of Protective Equipment

Effective use of protective equipment requires that the equipment be properly used, maintained, and inspected prior to use and periodically during the day. Site-specific issues and standard procedures will be reiterated during pre-entry training.

9.0 DECONTAMINATION

Equipment decontamination will be conducted to prevent cross-contamination during each sampling event. Sampling equipment will be properly cleaned according to the decontamination procedures discussed below.

Only clean, approved potable or deionized water will be used for equipment decontamination. Rinse water from equipment decontamination will initially be containerized, subsequently filtered through a carbon canister to remove VOCs, and drained onto the ground surface following filtration.

Submersible pumps will require decontamination prior to use and between use at each well location. Decontamination procedures are as follows:

- Steam clean pump and cables.
- Pump potable water through the pump. The potable water will be from a USACE-approved source.
- Pump dilute detergent water through the pump. The detergent water will be comprised of a teaspoon of non-phosphate detergent (such as Alconox) to one gallon of potable water.
- Rinse by pumping potable water through the pump.
- Spray the outside of the pump, screen, and impellers with 1% nitric acid.
- Rinse by spraying distilled, deionized water on the outside of the pump and screen section where the impellers are housed.
- Spray the outside of the pump, screen, and impellers with methanol
- Perform a final rinse by pumping distilled, deionized water through the pump.
- Air dry

New Teflon-lined polyethylene tubing will be dedicated for use in each well. Therefore, tubing decontamination is not required.

Decontamination of the interface probe (IFP) (or water level indicator) will be completed by cleaning with non-phosphate detergent followed by thoroughly rinsing with de-ionized water. IFPs are not required to follow the decontamination procedure steps discussed above for submersible pumps.

10.0 EMERGENCY RESPONSE PLAN

Prior to work start-up, personnel will be familiar with this Emergency Response Plan. The Site Manager will make this plan available for inspection and copying by subcontractors and will review the location of evacuation areas and exit routes.

Emergency telephone numbers will be carried by the field personnel at all times. See Table 5 for a listing of emergency telephone numbers.

Before commencing on-site operations, the ERT SSHO or his designee will advise personnel of the appropriate response to potential emergencies. Factors to be discussed will include the following.

- Communication networks and warning signals used on site;
- First aid equipment;
- Notification of fire, police and emergency facilities; and
- Evacuation plans.

10.1 Communication

The ERT team sampling members shall be equipped with a cellular telephone. If an emergency occurs, and the team members are not in close proximity to each other, communication will occur via telephone.

10.2 Pre-Planning

Pre-planning in case of an emergency helps minimize the potential hazards of an unpredictable event, such as physical injury. An emergency condition is considered to be present in the following instances:

- A member of the crew is involved in an accident or is experiencing adverse effects; and
- When a condition is discovered that suggests that a situation is more hazardous than first thought or anticipated.

Prior to conducting any activity, the ERT SSHO or his designee will implement the following general emergency procedures:

- Cellular telephones will be fully operable and turned on;
- Appropriate key personnel and emergency numbers will be carried by each field staff individual;
- Any accidents will be reported immediately to the ERT Project Manager and/or ERT SSHO so that appropriate actions can be taken;
- Personnel not familiar with certain operations will practice the operation prior to doing the actual work in the field; and
- The address of the nearest local hospital address will be noted (see Attachment 4, for a map of the hospital route and directions).

Table 10-1. Emergency Contact Information

Emergency Contact/Notification System		
Organization	**Agency**	**Telephone**
Ambulance	Local Emergency Services	911
Police	Local Emergency Services	911
Fire	Local Emergency Services	911
Hospital 1	Savanna Family Care Clinic	(815) 273-7110
Poison Control Center	Mid-Plains Poison Control Center	(800) 642-9999

10.3 Emergency Incident Procedures

In the event of an emergency, the information available at that time will be properly evaluated and the appropriate steps will be taken to implement the emergency response plan. The Site Manager will assume command of the situation. He/She will call the appropriate emergency services, evacuate personnel to the designated evacuation location as needed, and take other steps necessary to gain control over the emergency.

In the event of an emergency, the information available at that time will be properly evaluated and the appropriate steps will be taken to implement the emergency response procedures. The Site Manager (or SSHO if the Site Manager is part of the emergency) will assume command of the situation. He or she will call the appropriate emergency services, evacuate personnel to the pre-designated evacuation location as needed, and take other steps necessary to gain control over the emergency.

Give the following information when reporting an emergency:

1. Name and location of person reporting
2. Location of accident/incident
3. Name and affiliation of injured party
4. Description of injuries, fire, spill, or explosion
5. Status of medical aid and/or other emergency control efforts
6. Details of chemicals involved
7. Summary of accident, including suspected cause and time it occurred
8. Temporary control measures taken to minimize further risk

This information will not be released to parties other than those listed in this section and emergency response team members. Once emergency response agencies have been notified, the Project Manager will be notified immediately.

10.4 Emergency Notification Procedures

Emergency telephone numbers and directions to the nearest hospital are provided in Attachment 4, along with maps showing the routes to the nearest hospitals. The field personnel will immediately stop work and report to the Site Manager under the following potential emergency situations:

- Medical emergency
- Discovery of unanticipated hazards [e.g., drums, heavily contaminated materials, etc.]
- Overexposure of personnel to onsite contaminants
- Cold/heat-related injury or heat stress

Onsite emergencies may ultimately be handled by offsite emergency support personnel. Initial response and first-aid treatment, however, will be available through onsite personnel. In case of a hazardous materials emergency, the Site Manager will assume control and direction of the emergency response until arrival of offsite emergency personnel. The Site Manager will work with the SSHO to identify and evaluate hazards. Emergency responders and communications will be coordinated and controlled through the Site Manager.

When an incident is determined to be reportable to the USACE, the local USACE representative will be notified verbally within 4 hours. In addition, the incident shall be reported within five (5) working days on ENG Form 3394 to the local USACE representative, the USACE Project Technical Manager, and the NAB Safety and Occupational Health Office. Reportable accidents/incidents are defined as:

- Injuries to personnel resulting in lost work days;
- Non-lost work day cases which require medical treatment;
- Fire or explosion of any magnitude;
- Exposure of unprotected personnel to toxic agents;
- Property damage exceeding $2,000.00; and
- Any incidents involving the public or public property.

All injuries, illnesses, or high loss potential occurrences, no matter how minor they appear, are to be reported to the PM and/or SSHO, who will then ensure that the incident is logged, properly reported, and investigated. OSHA Logs (Attachment 7) will be completed for all incidents related to this project. OSHA recordable injuries and/or illnesses must be logged within three (3) days and forwarded to ERT's HR Director.

Under no circumstances should an injured employee drive themselves to the hospital, clinic etc. An employee with a minor injury may be transported by car after first aid treatment is provided on site. The person who transports the injured person should be trained in First Aid and CPR whenever possible. In the event the injury is severe, or when in doubt of severity of injury, the employee should be transported by ambulance.

Injured employees that require medical treatment or have been taken to a doctor, hospital, clinic, etc., should not be allowed to resume work without a written statement from the treating physician. This statement should provide a diagnosis, date of return to work, and work limitations. Should a statement such as "light duty" be provided, the SSHO will call the treating physician to determine the exact restriction. The SSHO will inform the treating physician of the type of work the employee normally performs and describe alternate work that is available to meet these work restrictions.

10.5 Personnel Injury/Medical Emergency

Personnel will always be alert for signs and symptoms of illnesses related to chemical, physical, and disease factors onsite. Severe injuries resulting from accidents will be recognized as emergencies and treated as such.

In a medical emergency, the Site Manager will cease work, and personnel will move to the decontamination area. Personnel currently trained in Standard First Aid/Adult CPR will evaluate the nature of the injury, decontaminate the victim if the victim can be moved safely, and initiate first aid assistance immediately. First aid will be administered as appropriate. The local Emergency Medical Services will be notified immediately if needed, contact numbers are provided in Attachment 4. Victims who are heavily contaminated with toxic or dangerous materials will be decontaminated before being transported from the site. A fellow worker will accompany injured workers to the hospital to inform the admitting clerk that the injury is work related and to assist in completing the insurance forms.

The Site Manager will complete an ERT Incident and Injury Report (Attachment 5) which will be submitted to the Project Manager and Health and Safety Manager within 24 hours of the following types of incidents:

- Job-related injuries and illnesses
- Accidents resulting in significant property damage
- Accidents involving vehicles and/or vessels
- Accidents in which there may have been no injury or property damage, but which have a high probability of recurring with at least a moderate risk to personnel or property

An accident, which results in a fatality or the hospitalization of 3 or more employees, will be reported within 8 hours to the U.S. Department of Labor by the Project Manager. Engineering Form 3394 (Attachment 6) will be submitted to the USACE Project Technical Manager within 48 hours of the accident. An Incident Report (Attachment 6) will be completed when a near miss occurs that could have potentially resulted in serious physical harm.

10.5.1 Incident Deterrence

In an effort to prevent incidents from occurring at ERT worksites, an effort will be made by all ERT employees to follow safe practices to eliminate any accidents. The inspection of sites by the SSHO and or the UXOSO on a daily basis will be one part of this effort. Other methods of reducing accidents will include vigorously investigating any high loss potential ("near-miss") incidents. This will include a causal analysis to determine the immediate and basic causes. The information obtained in the causal analysis will be used to develop appropriate corrective actions to prevent the recurrence of similar incidents.

10.5.2 High-Loss Potential Incident Reporting

In the daily operations, all reporting of high-loss potential incidents will be encouraged and will be investigated by the supervisor, SSHO, and other employees as needed for proactive remediation. Furthermore, the reports will be forwarded to ERT's Human Resource Director where trends within the company can be tracked to present similar high loss potential incidents from occurring at other ERT projects.

10.5.3 Incident Reporting and Investigation

All incidents will be immediately reported to the Supervisor and the UXOSO or SSHO. The SSHO will record all applicable incidents/injuries/near misses. If required, emergency medical care or first aid will be rendered.

As soon as possible after the occurrence of an occupational incident, but no later than 24 hours, the supervisor, with the assistance of the SSHO, will initiate an incident investigation. The incident investigation will be documented on an Incident Investigation Report. The purpose of the incident investigation is to identify the causes of the incident in order to take corrective action to prevent future occurrences.

Reporting of all incidents will be made to ERT's Human Resources Director, who maintains a log of all the recordable injuries and illnesses. Refer to Attachment 7 which contains a copy of ERT's Incident and Injury Reporting form and the OSHA Log 301 Form.

11.0 REFERENCES

Occupational Safety and Health Administration (OSHA), 2004. *Hazardous Waste Operations and Emergency Response*, Regulation 29 CFR 1910.120

OSHA, 2006. *Safety and Health for Construction*, Regulation 29 CFR 126

USACE 2003. *Safety and Health Requirements*, Engineering Manual 385-1-1.

USACE 2004a. *Munitions and Explosives of Concern (MEC) Support During Hazardous, Toxic, and Radioactive Waste (HTRW) and Construction Activities Engineer Pamphlet* EP 75-1-2.

U.S Army Corps of Engineers (USACE), 2004b. *Basic Safety Concepts and Considerations for Munitions and Explosives of Concern (MEC) Response Action Operationss* (EP 385-1-95a).

Attachment 1
Health and Safety Report Forms

ERT Vehicle Inspection Form (Trucks)

DATE: _____ **TIME:** _____

This inspection form is to be filled out at the start of the work shift upon deliveries by the Equipment/Truck Operator to insure that the equipment/truck is safe to operate and is free from apparent damage, which could cause failure while in use. Once completed, this form is to be given to the Site Superintendent or Safety Officer to be kept on file on-site. In all cases, consult the manufacturer's data to ensure compliance with all inspection criteria, which may not be indicated below.

Make/Description: _____ Model: _____

EQUIPMENT	PASS	FAIL	NA	COMMENTS AND ACTION TAKEN
Brakes				
Brake lights				
Reverse signal alarm				
Horn/Air horn				
Tires/Tracks				
Steering				
Seat belts				
Operating controls				
Fire extinguisher				
Lights				
Defroster				
Mirrors				
Instruments				
Coupling devices				
Windshield/window glass				
Mud flaps/rock guards				
Exhaust system				
Hitches and safety cables				
Hydraulic lines/air hoses				
Engine oil level				
Roll over equipment				

ODOMETER: _____ **HOUR METER:** _____ **FUEL LEVEL:** _____

INSPECTOR'S NAME AND SIGNATURE: _____

SITE SUPERINTENDENT'S NAME AND SIGNATURE: _____

(Superintendent must sign off prior to operating a vehicle with a noted deficiency, if not corrected immediately)

ERT SELF EVALUATION CHECKLIST

The purpose of the ERT Self Evaluation Inspection Form is to identify and correct problems so as to avoid project delays. ERT's Self Evaluation can determine conditions that need to be corrected or improved to bring operations up to acceptable standards, both from safety and operational standpoints. Secondary purposes are to improve operations and thus to increase efficiency, effectiveness, and profitability.

Project: **Date**:

Inspection Type: ❑ Daily ❑ Weekly ❑ Monthly ❑ Corporate

Area(s) of Inspections:

Evaluation Conducted By:

CATEGORY	OBSERVATIONS (N/A if not applicable)	RECOMMENDATIONS
RECORD KEEPING		
1. OSHA Postings		
2. Employee Safety Training		
3. Medical Surveillance Program		
4. Site Safety & Health Plan Sign-off		
5. Exposure Monitoring Records		
6. Daily Tailgate Safety Meetings		
7. Visitors Sign-Off		
8. Accident Investigation Reports		
9. Workers Compensation Claims (Please indicate claimant's name/date of incident)		

CATEGORY	OBSERVATIONS (N/A if not applicable)	RECOMMENDATIONS
GENERAL WORKPLACE		
1. House Keeping		
2. Noise Exposure		
3. Lighting/Illumination		
4. Field/Office Ergonomics		
5. Roadways/Traffic Control		
6. Scaffolding/Fall Protection		
7. Sanitation/Toilet/Wash Facilities		
HAZARD COMMUNICATION		
1. List of Hazardous Materials		
2. Hazardous Materials Labeling		
3. Material Safety Data Sheets		
4. Employee Training		
EQUIPMENT OPERATIONS		
1. Inspection Checklists (Heavy Equipment)		
2. Hand Portable Tools Inspections		
3. Machine/Equipment Guarding		
4. Hoisting/Rigging Operations		
HAZARD CONTROLS		
1. Lockout/Tagout Systems		
2. Site Control (EZ, CRZ, SZ)		
3. Decontamination Procedures		
4. Safety Signs		
5. Site Security		
EMPLOYEE PROTECTIVE EQUIPMENT		
1. Hearing Protection		

ERT Visitor Information Form

You are visiting a project site where Unexploded Ordnance (UXO) is known to be present. Exposure to this hazard presents a danger of injury due to unintentional detonation of UXO. You will remain with your designated escort at all times and follow his/her instructions for your safety and the safety of others. You must also wear all protective clothing and equipment issued. Minimum protective equipment will be Level D protection (safety work boots and safety glasses). Equipment issued must be returned prior to leaving the site.

Visitor Certification

I acknowledged that I have been advised of the dangers present at this project site. I agree to immediately follow all direction(s) given by my escort on site. I also certify that I do relieve ERT, the U.S. Government, the applicable state which the project site is located, their officers, employees, and agents of all liability of all consequences raising from and related to the potential hazards associated with entry to this site.

Name	Company	Date

Earth Resources Technology, Inc.

Site Health and Safety Plan

Compliance Agreement

I have received a copy of the Site Specific Safety and Health Plan for the project. I have received information and training on the contents of the plan including operations to be performed, site hazards, safety requirements, use of personal protective clothing and equipment, monitoring requirements, site control, and decontamination procedures and actions to take in the event of a site emergency. I have reviewed the plan, understand its requirements, and agree to comply with all of its provisions. I understand that failure to comply with these requirements could result in disciplinary action.

Signature	**Position**	**Employer**	**Date**

EARTH RESOURCES TECHNOLOGY
ACCIDENT INVESTIGATION REPORT

Location: _____ Department: _____

Name of Injured	Social Security Number	Sex ☒M ☐F	Age	Date of Accident
Home Address of Injured _____ _____				

Employee's Usual Occupation	Occupation at time of Accident
Length of Employment ☐Less than 1 month ☐1-5 months ☐6 months - 5 years ☐More than 5 years	Employment Category ☐Regular, full-time ☐Temporary ☐Regular, part-time ☐Seasonal ☐Non-employee

Name and Address of Physician	Name and Address of Hospital
_____ _____ _____ _____	_____ _____ _____ _____

Names of Others Injured in the Same Accident

1._____ 2._____ 3._____

4._____ 5._____ 6._____

Nature of Injury and Part of Body Injured
Severity of Injury ☐Fatality ☐Medical treatment ☐Days away from work – How many? _____ ☐First Aid ☐Restricted Activity – How many days? _____ ☐Other – Please specify: _____

Time of Injury	Shift on duty at time of Accident	Specific Location of Accident On employer's premises? ☐ Yes ☐ No

How many hours had injured worker been on duty when accident occurred?

Phase of Employee's Workday at Time of Injury

☐ Rest Period ☐ Meal Period ☐ Regular Work Time
☐ Overtime ☐ Entering/Leaving site
☐ Other – Please Specify: _____

Supervision at time of Accident

☐ Direct ☐ Indirect ☐ Unsupervised
☐ Supervision not feasible – Please explain:

_____

Task and Activity at time of Accident

General Type of Task: _____

Specific Activity: _____

Employee was working: ☐ Alone ☐ With crew or co-worker ☐ Other – Please Specify:

_____

Describe how the Accident Occurred:

Describe Events and Conditions that Contributed to the Accident:

Note: ECC Senior Management personnel shall review this Accident Investigation Report. ECC management may request a drug screening test for the individual(s) directly involved in a work related accident involving an injury, death, property damage or vehicle/equipment damage.

Prepared by: _____ Title: _____
Dept: _____ Date: _____

Attachment 2
Material Safety Data Sheets

MATERIAL SAETY DATA SHEET
(HYDROCHLORIC ACID)

I. PRODUCT IDENTIFICATION

Chemical Name : Hydrochloric Acid
Trade Name : Technical Grade Muriatic Acid
Synonyms : Muriatic Acid, Spirit of Salts

II. COMPOSITION /INGREDIENTS

Hydrochloric Acid, % : 32 – 34 % by weight
Chemical Formula : HCl
Molecular Weight : 36.46 g/mole
CAS Registry No. : 7647-01-0

III. HAZARDS IDENTIFICATION

THIS PRODUCT MAY BE : corrosive, toxic and a major potential hazard upon contact to skin, eyes and respiratory tract.

TOXICITY ROUTES OF EXPOSURE :

Ingestion can cause severe burns of the mucous membranes of the mouth, esophagus and stomach; pain, nausea and vomiting may also occur.

Inhalation causes irritation of the upper respiratory tract resulting in cough, burning of the throat and choking sensation.

Skin contact to a high concentration of the HCl gas or liquid may cause burns; repeated or prolonged exposures to dilute solutions may cause dermatitis.

Eye contact to high concentration of the acid can cause eye irritation to severe destruction like prolonged or permanent visual impairment, including blindness. These effects occur rapidly affecting all parts of the eye. Mist can also cause irritation to destructive burns.

OVEREXPOSURE :

Can cause serious damage to all body tissues contacted.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:

Fumes may aggravate eye, skin or respiratory conditions. Effects are usually limited to inflammation and occasionally ulceration of the nose, throat and larynx, if inhaled deeply, pulmonary edema may occur.

IV. FIRST AID MEASURES

SKIN : Remove contaminated clothing and immediately wash skin for a minimum of 15 minutes. Call or see a physician.

EYES : Immediately flush eyes with large amount of water. Occasionally lifting the upper and lower eyelids and rotating the eyeballs. Continue flushing for a minimum of 15 minutes. Call a physician.

INHALATION : Remove to fresh air. If breathing stops, administer artificial respiration. Call a physician.

INGESTION : DO NOT induce vomiting. Rinse or wash mouth with water. If person is conscious, give 2 or more glasses of water. If unconscious, never give anything by oth**Se a pla**ntly.

V. FIRE FIGHTING MEASURES

Autoignition Point : Not Applicable
Flash Point : Not Applicable
Flammability/Explosive limits : Not Applicable
Fire/Explosion Hazards: Emits toxic and choking fumes of hydrogen chloride. Hydrochloric acid is not flammable but flammable and explosive hydrogen gas may be formed on contact with metals.
Fire Prevention/ Extinguishing Media : Not Applicable

VI. ACCIDENTAL RELEASE ME ASURES

IN CASE OF SPILL OR RELEASE ;

Move people from the area. Move upwind. Avoid contact with acid. Stop leaks if safe to do so. Reposition container if this will reduce or stop leakage. If leak continues, remove leaking container from vehicle or move other materials from vehicle away from container. Absorb spill with sand or earth. If available, cover the spill with excess soda ash, lime or sodium bicarbonate, otherwise, wash away with large amounts of water. Scoop slurry to plastic drums. If leak cannot be safely stopped or if contents cannot be safely transferred to a sound container, contact fire brigade.

VII. HANDLING AND STORAGE

Storage Requirements: Keep container tightly closed.
FOR SMALL VOLUMES : Maybe stored in plastic jugs, carboys, and plastic drums.
FOR LARGE VOLUMES : Store in rubber-lined or epoxy lined steel storage tanks or fiber glass reinforced polyester (FRP) tanks.
Incompatible Materials: Store away from heat
Use Instructions: Wear suitable protective clothing, gloves and eye/face protection. In case of insufficient ventilation, wear suitable respiratory equipment.

MATERIAL SAETY DATA SHEET
(HYDROCHLORIC ACID)

VIII. EXPOSURE CONTROLS AND PROTECTION

Ventilation: Use only in well-ventilated areas.
Protective Equipment for the eyes and skin :
Splash proof and face shield goggles, disposable latex/ rubber apron, PVC rain suit, rubber boots with pant legs over boots.
Respiratory Protection Requirements: NIOSH/MSHA approved respirator should be used.
Precautionary Hygiene/control measures :
Avoid contact with skin, eyes, and clothing. Do not breathe mist or vapor. Wash thoroughly after handling. Safety showers and eye wash fountains should be available in storage and handling area. Any protective clothing contaminated with hydrochloric acid should be removed immediately and thoroughly laundered before wearing again.

IX PHYSICAL AND CHEMICAL PROPERTIES

STATE	:	fuming liquid
APPEARANCE	:	colorless to slightly yellow
ODOR	:	Irritating
pH	:	Strong acid <1
BOILING POINT	:	85^0 C
FLASH POINT	;	Not determined
SPECIFIC GRAVITY	:	1.150 -1.164
VAPOR PRESSURE	:	20 hPa @ 20^0 C
SOLUBILITY IN	:	WATER: miscible, BASE : miscible

X STABILITY AND REACTIVITY

Stability : Stable under normal handling conditions.
Hazardous polymerization will not occur.
Hazardous decomposition product: HCl gas will not decompose.
Materials and conditions to avoid (incompatibility) are:
Avoid high temperatures. Containers may burst. Corrosive to most metals, concrete, some plastics, some rubber and coatings. Fumes forms droplets which settle and promote corrosion of metals and unprotected equipment. Mixing with strong acids can cause evolution of hydrogen chloride gas. Oxidizing agents will cause the release of toxic chlorine gas. Contact of liquid acid or gas with alkali or active metal may develop enough heat to cause fire in adjacent combustible material.

XI TOXICOLOGICAL INFORMATION

Reproductive Effects: No data available
MUTAGENICITY : Not applicable
CANCER INFORMATION : Not applicable

XII. ECOLOGICAL INFORMATION

ECOTOXICITY DATA: High acidity may pose potential hazard to plant and marine life.
WATER-POLLUTION RISK CLASSIFICATION: Slightly water-polluting substance.

XIII. DISPOSAL CONSIDERATIONS

Dispose of in accordance with all Government and Local regulations.

XIV. TRANSPORT INFORMATION

Transportation of Dangerous Goods
TDG Classification: Do not ship by air.
DOT Hazard Classification: Class 8 : Corrosive: Group II
DOT Shipping Name : Hydrochloric acid ID: UN 1789

XV. REGULATORY INFORMATION

No data available

XVI OTHER INFORMATION

This MSDS contains information under the sixteen (16) section headings required by ISO 11014 "Safety Data Sheet for Chemical Products".

THE INFORMATION CONTAINED HEREIN IS PRESENTED IN GOOD FAITH AND BELIEVED TO CORRECT AS OF THE DATE OF ISSUE. HOWEVER, NO WARRANTY, EXPRESS OR IMPLIED IS GIVEN BY MABUHAY VINYL CORPORATION REGARDING THE USE OF THIS MATERIAL SAFETY DATA SHEET (MSDS).

MSDS **Material Safety Data Sheet**

From: Vinquiry, Inc.
7795 Bell Road
Windsor, CA 95492

VINQUIRY

All Non-[illegible]

24 hour Emergency Telephone:
Chemtrec: 1-800-424-9300

Outside U.S. and Canada Chemtrec: 202-483-7616

NOTE: CHEMTREC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

Sulfuric Acid 1+3

SULFURIC ACID, 10 - 51%

MSDS Number: SA264 --- *Effective Date: 01/01/01*

1. Product Identification

Synonyms: Oil of vitriol; Babcock acid; sulphuric acid
CAS No.: 7664-93-9
Molecular Weight: 98.07
Chemical Formula: H2SO4 in H2O
Vinquiry Inc. Product Codes: 10-264-0000, 10-264-0118, 10-264-0237, 10-264-0473, 10-264-0946

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Sulfuric Acid	7664-93-9	10 - 51%	Yes
Water	7732-18-5	49 - 90%	No

3. Hazards Identification

Emergency Overview

POISON! DANGER! CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR CONTACTED WITH SKIN. HARMFUL IF INHALED. AFFECTS TEETH. WATER REACTIVE. CANCER HAZARD. STRONG INORGANIC ACID MISTS CONTAINING SULFURIC ACID CAN CAUSE CANCER. Risk of cancer depends on duration and level of exposure.

VINQUIRY INC. SAFETY DATA Ratings (Provided here for your convenience)

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Water Reactive)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Potential Health Effects

Inhalation:
Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. May cause lung edema, a medical emergency.
Ingestion:
Corrosive. Swallowing can cause severe burns of the mouth, throat, and stomach, leading to death. Can cause sore throat, vomiting, diarrhea. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow ingestion or skin contact. Circulatory shock is often the immediate cause of death.
Skin Contact:
Corrosive. Symptoms of redness, pain, and severe burn can occur. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow skin contact or ingestion. Circulatory shock is often the immediate cause of death.
Eye Contact:
Corrosive. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Chronic Exposure:
Long-term exposure to mist or vapors may cause damage to teeth. Chronic exposure to mists containing sulfuric acid is a cancer hazard.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

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4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician immediately.
Ingestion:
DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Call a physician immediately.
Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Excess acid on skin can be neutralized with a 2% solution of bicarbonate of soda. Call a physician immediately.
Eye Contact:

Immediately flush eyes with gentle but large stream of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Call a physician immediately.

5. Fire Fighting Measures

Fire:
Concentrated material is a strong dehydrating agent. Reacts with organic materials and may cause ignition of finely divided materials on contact.
Explosion:
Contact with most metals causes formation of flammable and explosive hydrogen gas.
Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Do not use water on material. However, water spray may be used to keep fire exposed containers cool.
Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Structural firefighter's protective clothing is ineffective for fires involving this material. Stay away from sealed containers.

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

NEUTRASORB(R) or TEAM(R) 'Low Na+' acid neutralizers are recommended for spills of this product.

7. Handling and Storage

Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it for other purposes. When diluting, always add the acid to water; never add water to the acid. When opening metal containers, use non-sparking tools because of the possibility of hydrogen gas being present. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
For Sulfuric Acid:

- OSHA Permissible Exposure Limit (PEL) -
1 mg/m3 (TWA)
- ACGIH Threshold Limit Value (TLV) -
1 mg/m3(TWA), 3 mg/m3 (STEL), A2 - suspected human carcinogen for sulfuric acid contained in strong inorganic acid mists.

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, a full facepiece respirator with an acid gas cartridge and particulate filter (NIOSH type N100 filter) may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P particulate filter. For emergencies or instances where the exposure levels are not known, use a full- facepiece positive-pressure, air-supplied respirator. WARNING: Air purifying respirators do not protect workers in oxygen-deficient atmospheres. Where respirators are required, you must have a written program covering the basic requirements in the OSHA respirator standard. These include training, fit testing, medical approval, cleaning, maintenance, cartridge change schedules, etc. See 29CFR1910.134 for details.

Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

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9. Physical and Chemical Properties

Appearance:
Clear oily liquid.

Odor:
Odorless.

Solubility:
Miscible with water, liberates much heat.

Specific Gravity:
1.40 (50%), 1.07 (10%)

pH:
1 N solution (ca. 5% w/w) = 0.3; 0.1 N solution (ca. 0.5% w/w) = 1.2; 0.01 N solution (ca. 0.05% w/w) = 2.1.

% Volatiles by volume @ 21C (70F):
No information found.

Boiling Point:
ca. 290C (ca. 554F) (decomposes at 340C)

Melting Point:
3C (100%), -32C (93%), -38C (78%), -64C (65%).

Vapor Density (Air=1):
3.4

Vapor Pressure (mm Hg):
1 @ 145.8C (295F)
Evaporation Rate (BuAc=1):
No information found.

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10. Stability and Reactivity

Stability:
Concentrated solutions react violently with water, spattering and liberating heat.
Hazardous Decomposition Products:
Toxic fumes of oxides of sulfur when heated to decomposition. Will react with water or steam to produce toxic and corrosive fumes. Reacts with carbonates to generate carbon dioxide gas, and with cyanides and sulfides to form poisonous hydrogen cyanide and hydrogen sulfide respectively.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Water, potassium chlorate, potassium perchlorate, potassium permanganate, sodium, lithium, bases, organic material, halogens, metal acetylides, oxides and hydrides, metals (yields hydrogen gas), strong oxidizing and reducing agents and many other reactive substances.
Conditions to Avoid:
Heat, moisture, incompatibles.

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11. Toxicological Information

Toxicological Data:
Oral rat LD50: 2140 mg/kg; inhalation rat LC50: 510 mg/m3/2H; standard Draize, eye rabbit, 250 ug (severe); investigated as a tumorigen, mutagen, reproductive effector.
Carcinogenicity:
Cancer Status: The International Agency for Research on Cancer (IARC) has classified "strong inorganic acid mists containing sulfuric acid" as a known human carcinogen, (IARC category 1). This classification applies only to mists containing sulfuric acid and not to sulfuric acid or sulfuric acid solutions.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Sulfuric Acid (7664-93-9)	No	No	None
Water (7732-18-5)	No	No	None

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12. Ecological Information

Environmental Fate:
When released into the soil, this material may leach into groundwater. When released into the air, this material may be removed from the atmosphere to a moderate extent by wet deposition. When released into the air, this material may be removed from the atmosphere to a moderate extent by dry deposition.
Environmental Toxicity:
LC50 Flounder 100 to 330 mg/l/48 hr aerated water/Conditions of bioassay not specified; LC50 Shrimp

80 to 90 mg/l/48 hr aerated water /Conditions of bioassay not specified; LC50 Prawn 42.5 ppm/48 hr salt water /Conditions of bioassay not specified.
This material may be toxic to aquatic life.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: SULFURIC ACID (WITH NOT MORE THAN 51% ACID)
Hazard Class: 8
UN/NA: UN2796
Packing Group: II
Information reported for product/size: 200L

International (Water, I.M.O.)

Proper Shipping Name: SULPHURIC ACID (WITH NOT MORE THAN 51% ACID)
Hazard Class: 8
UN/NA: UN2796
Packing Group: II
Information reported for product/size: 200L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Sulfuric Acid (7664-93-9)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Sulfuric Acid (7664-93-9)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\---------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.

Sulfuric Acid (7664-93-9)	1000	1000	Yes	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Sulfuric Acid (7664-93-9)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: Yes
SARA 311/312: Acute: Yes Chronic: Yes Fire: No Pressure: No
Reactivity: Yes (Pure / Liquid)

Australian Hazchem Code: 2P
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **2** Other: **Water reactive**
Label Hazard Warning:
POISON! DANGER! CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR CONTACTED WITH SKIN. HARMFUL IF INHALED. AFFECTS TEETH. WATER REACTIVE. CANCER HAZARD. STRONG INORGANIC ACID MISTS CONTAINING SULFURIC ACID CAN CAUSE CANCER. Risk of cancer depends on duration and level of exposure.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe mist.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Do not contact with water.
Label First Aid:
In all cases call a physician immediately. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before re-use. Excess acid on skin can be neutralized with a 2% bicarbonate of soda solution. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.
Product Use:
Laboratory Reagent.
Revision Information:

Disclaimer

Vinquiry Inc. provides this information in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to laboratory use of this material by a properly trained person. Individuals receiving this information must exercise their independent judgment in determining its appropriateness for a particular purpose. Vinquiry Inc. will not be responsible for damages resulting from use or reliance upon this information.

MSDS Number: **N3660** * * * * * *Effective Date:* ***02/15/08*** * * * * * *Supercedes:* ***05/06/05***

MSDS **Material Safety Data Sheet**

From: **Mallinckrodt Baker, Inc.**
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS | J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals.

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance.

NITRIC ACID, 50-70%

1. Product Identification

Synonyms: Aqua Fortis; Azotic Acid; Nitric Acid 50%; Nitric Acid 65%; nitric acid 69-70%
CAS No.: 7697-37-2
Molecular Weight: 63.01
Chemical Formula: HNO3
Product Codes:
J.T. Baker: 5371, 5796, 5801, 5826, 5856, 5876, 5896, 9597, 9598, 9600, 9601, 9602, 9603, 9604, 9606, 9607, 9608, 9610, 9616, 9617, 9670
Mallinckrodt: 1409, 2704, 2705, 2716, 6623, H862, H988, H993, H998, V077, V650

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Nitric Acid	7697-37-2	50 - 70%	Yes
Water	7732-18-5	30 - 50%	No

3. Hazards Identification

Emergency Overview

POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.

SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 4 - Extreme (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 4 - Extreme (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Potential Health Effects

Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

Inhalation:
Corrosive! Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract.
Ingestion:
Corrosive! Swallowing nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract.
Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color.
Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth and lung damage. Long-term exposures seldom occur due to the corrosive properties of the acid.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders, eye disease, or cardiopulmonary diseases may be more susceptible to the effects of this substance.

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4. First Aid Measures

Immediate first aid treatment reduces the health effects of this substance.
Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

---

5. Fire Fighting Measures

Fire:
Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition. Can react with metals to release flammable hydrogen gas.
Explosion:
Reacts explosively with combustible organic or readily oxidizable materials such as: alcohols, turpentine, charcoal, organic refuse, metal powder, hydrogen sulfide, etc. Reacts with most metals to release hydrogen gas which can form explosive mixtures with air.
Fire Extinguishing Media:
Water spray may be used to keep fire exposed containers cool. Do not get water inside container.
Special Information:
Increases the flammability of combustible, organic and readily oxidizable materials. In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

---

6. Accidental Release Measures

Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash, lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker NEUTRASORB® acid neutralizers are recommended for spills of this product.

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7. Handling and Storage

Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it for other purposes. When diluting, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

---

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:

-OSHA Permissible Exposure Limit (PEL):
2 ppm (TWA), 4 ppm (STEL)
-ACGIH Threshold Limit Value (TLV):
2 ppm (TWA); 4 ppm (STEL)

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.
Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Nitric acid is an oxidizer and should not come in contact with cartridges and canisters that contain oxidizable materials, such as activated charcoal. Canister-type respirators using sorbents are ineffective.
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

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9. Physical and Chemical Properties

Appearance:
Colorless to yellowish liquid.
Odor:
Suffocating, acrid.
Solubility:
Infinitely soluble.
Specific Gravity:
1.41
pH:
1.0 (0.1M solution)
% Volatiles by volume @ 21C (70F):
100 (as water and acid)
Boiling Point:
122C (252F)
Melting Point:
-42C (-44F)
Vapor Density (Air=1):
2-3
Vapor Pressure (mm Hg):
48 @ 20C (68F)
Evaporation Rate (BuAc=1):
No information found.

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10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Containers may burst when heated.

Hazardous Decomposition Products:
When heated to decomposition, emits toxic nitrogen oxides fumes and hydrogen nitrate. Will react with water or steam to produce heat and toxic and corrosive fumes.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
A dangerously powerful oxidizing agent, concentrated nitric acid is incompatible with most substances, especially strong bases, metallic powders, carbides, hydrogen sulfide, turpentine, and combustible organics.
Conditions to Avoid:
Light and heat.

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11. Toxicological Information

Nitric acid: Inhalation rat LC50: 244 ppm (NO2)/30M; Investigated as a mutagen, reproductive effector. Oral (human) LDLo: 430 mg/kg.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Nitric Acid (7697-37-2)	No	No	None
Water (7732-18-5)	No	No	None

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12. Ecological Information

Environmental Fate:
No information found.
Environmental Toxicity:
No information found.

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13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be managed in an appropriate and approved waste facility. Although not a listed RCRA hazardous waste, this material may exhibit one or more characteristics of a hazardous waste and require appropriate analysis to determine specific disposal requirements. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

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14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: NITRIC ACID
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 6.5GL

International (Water, I.M.O.)

Proper Shipping Name: NITRIC ACID (WITH NOT MORE THAN 70% NITRIC ACID)
Hazard Class: 8
UN/NA: UN2031
Packing Group: II
Information reported for product/size: 6.5GL

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15. Regulatory Information

--------\Chemical Inventory Status - Part 1\---------------------------------

Ingredient	TSCA	EC	Japan	Australia
Nitric Acid (7697-37-2)	Yes	Yes	Yes	Yes
Water (7732-18-5)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\---------------------------------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Nitric Acid (7697-37-2)	Yes	Yes	No	Yes
Water (7732-18-5)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Nitric Acid (7697-37-2)	1000	1000	Yes	No
Water (7732-18-5)	No	No	No	No

--------\Federal, State & International Regulations - Part 2\----------------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Nitric Acid (7697-37-2)	1000	No	No
Water (7732-18-5)	No	No	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

Australian Hazchem Code: 2PE
Poison Schedule: S6
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

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16. Other Information

NFPA Ratings: Health: **3** Flammability: **0** Reactivity: **0** Other: **Oxidizer**
Label Hazard Warning:
POISON! DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG AND TOOTH DAMAGE.
Label Precautions:
Do not get in eyes, on skin, or on clothing.
Do not breathe vapor or mist.
Use only with adequate ventilation.
Wash thoroughly after handling.
Keep from contact with clothing and other combustible materials.
Do not store near combustible materials.
Store in a tightly closed container.
Remove and wash contaminated clothing promptly.
Label First Aid:
In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In all cases get medical attention immediately.
Product Use:
Laboratory Reagent.
Revision Information:
No Changes.
Disclaimer:
****
Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.
****
Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

MATERIAL SAETY DATA SHEET
(CAUSTIC SODA)

I. PRODUCT IDENTIFICATION

Chemical Name : Sodium Hydroxide
Trade Name : Caustic Soda, 50 %
Synonyms : Liquid Caustic Soda, Caustic, Soda Lye, Lye Solution

II. COMPOSITION /INGREDIENTS

Sodium Hydroxide, % : 48 – 52 % by weight
Chemical Formula : NaOH
Molecular Weight : 40 g/mole
CAS Registry No. : 1310-73-2

III. HAZARDS IDENTIFICATION

THIS PRODUCT MAY BE : corrosive, toxic and a major potential hazard upon contact to skin and eyes.
TOXICITY ROUTES OF EXPOSURE : Ingestion can cause severe burning and pain in lips, mouth, tongue, throat and stomach. Death can result from ingestion.
OVEREXPOSURE : Causes burns and scarring. Can cause serious damage to all body tissues contacted.

CANCER INFORMATION : Not applicable
MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Chronic eye or skin conditions

IV. FIRST AID MEASURES

SKIN : Remove contaminated clothing and immediately wash skin for a minimum of 15 minutes. Call or see a physician.
EYES : Immediately flush eyes with large amount of water, occasionally lifting the upper and lower eyelids and rotating the eyeballs. Continue flushing for a minimum of 15 minutes. See a physician.
INHALATION : Remove to fresh air. If breathing stops, administer artificial respiration. See a physician.
INGESTION : DO NOT induce vomiting. If person is conscious, give 2 or more glasses of water. If unconscious, never give anything by mouth. See a physician immediately.

V. FIRE FIGHTING MEASURES

Autoignition Point : Not Applicable
Flammability/Explosive limits : Not Applicable
Fire/Explosion Hazards: Contact with strong acids may generate enough heat to ignite combustibles.
Fire Prevention : Not Applicable

VI. ACCIDENTAL RELEASE MEASURES

IN CASE OF SPILL OR RELEASE : Completely contain spilled material with dikes, sandbags, etc., and prevent run off into the ground or surface waters or sewers. Recover as much caustic material as possible into containers for disposal. Add water and neutralize remaining caustic material with dilute hydrochloric acid, citric acid or another solid acidic material to a pH between 6 and 9. Collect neutralized caustic with a dry sorbent. Flush residual neutralized waste to the drain with excess water.

VII. HANDLING AND STORAGE

Storage Requirements: Keep container tightly closed.
FOR SMALL VOLUMES : Maybe stored in plastic jugs.
FOR LARGE VOLUMES ; Store in steel storage tanks.
INCOMPATIBLE MATERIALS : Store away from acids. (Refer to Section X)

VIII. EXPOSURE CONTROLS AND PROTECTION

Adequate ventilation needed. TLV C : 2 mg/m^3
Protective Equipment for the eyes and skin :
Goggles, respirator, disposable latex/ rubber apron, PVC rain suit, rubber boots with pant legs over boots.
Precautionary Hygiene/control measures :
Avoid contact with skin, eyes, and clothing.
Do not breathe mist or vapor. Wash thoroughly after handling. Safety showers and eye wash fountains should be available in storage and handling area.

IX PHYSICAL AND CHEMICAL PROPERTIES

STATE	:	liquid
APPEARANCE	:	colorless or slightly turbid
ODOR	:	Irritating
pH	:	Strong base >14
BOILING POINT	:	145 ^0C for ~50% NaOH Solution
FLASH POINT	;	Not determined
SPECIFIC GRAVITY	:	1.51-1.54
VAPOR PRESSURE	:	~6.3 mm Hg @ 40^0C
SOLUBILITY IN	:	WATER: miscible, ACID : miscible

MATERIAL SAETY DATA SHEET
(CAUSTIC SODA)

X STABILITY AND REACTIVITY

Stable under normal handling conditions. Materials and conditions to avoid (incompatibility) are:

- Chlorinated hydrocarbons, acetaldehyde, acrolein, aluminum, chlorine triflouride, hydroquinone, maleic anhydride, and phosphorous pentoxide.
- Dilution with water evolves large quantity of heat.

Hazardous decomposition & combustion product = none
Hazardous polymerization will not occur.

IX TOXICOLOGICAL INFORMATION

Effects from skin contact – Contact with skin can cause severe burns with deep ulcerations. Contact with solution or mist can cause multiple burns with temporary loss of hair at burn site.

Effects from eye contact – Liquid in the eye can cause severe destruction and blindness. These effects can occur rapidly affecting all parts of the eye. Mist can cause irritation with high concentration causing destructive burns.

IX. ECOLOGICAL INFORMATION

ECOTOXICITY DATA : High basicity may pose potential hazard to plant and marine life.

XII. DISPOSAL CONSIDERATIONS

Dispose of in accordance with all Government and Local regulations.

XIV. TRANSPORT INFORMATION

Transportation of Dangerous Goods
TDG Classification: Do not ship by air.
DOT Hazard Classification: Class 8 : Corrosive
DOT Shipping Name : Sodium Hydroxide ID: UN1824

XV. REGULATORY INFORMATION

No data available

XVI OTHER INFORMATION

This MSDS contains information under the sixteen (16) section headings written in accordance with the International Standard ISO 11014 *"Safety Data Sheet for Chemical Products".*

THE INFORMATION CONTAINED HEREIN IS PRESENTED IN GOOD FAITH AND BELIEVED TO BE CORRECT AS OF THE DATE ISSUED. HOWEVER, NO WARRANTY, EXPRESSED OR IMPLIED, IS GIVEN BY MABUHAY VINYL CORPORATION REGARDING THE USE OF THIS MATERIAL SAFETY DATA SHEET (MSDS).

Attachment 3
Activity Hazard Analyses

ACTIVITY HAZARD ANALYSIS

Activity: General Site Preparation and Security
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
Implement security and access control measures	Unauthorized personnel and the endangerment of wildlife.	? Provide security measures (e.g., fencing, and barricades) to prevent unauthorized entry. Incorporate access control measures to preserve wildlife.
General site preparation	Slips, trips, and falls	? Hazards will be identified and remedied by implementation of engineering controls. Good housekeeping procedures shall be enforced at all times. Continual inspection of all work areas.
	Cold Stress	? Take rest breaks in warm shelter ? Drink warm fluids throughout the day. ? Wear insulated clothing. ? Buddy system shall be utilized. ? Adjust work schedule when needed.

PPE/SAFETY SUPPLIES	SAFETY INSPECTIONS	TRAINING
Level D PPE (hard hat, steel-toed boots, eye protection and general work clothes) + Reflector vests and hearing protection (when applicable).	Heavy Equipment (upon arrival and prior to usage) General Site Safety (Daily)	AHA Review "Mobilization and Site Preparation" HAZWOPER Certification (40-hr, 8-hr) OSHA Supervisory (Required for

ACTIVITY HAZARD ANALYSIS

Activity: Soil Sampling
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPAL STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
Mobilization of sampling supplies/equipment	Lifting	Use proper lifting techniques; Wear appropriate personal protective equipment. Objects greater than 60 lbs. require assistance or use of a mechanical lifting device.
Implement site security and access control measures	Unauthorized personnel and the endangerment of wildlife.	Provide security measures (e.g., fencing, and barricades) to prevent unauthorized entry. Incorporate access control measures to preserve wildlife.
General sampling activities	Slips, trips, and falls near excavation Cold Stress	Hazards will be identified and remedied by implementation of engineering controls. Good housekeeping procedures shall be enforced at all times. Continual inspection of all work areas. Take rest breaks in warm shelter Drink warm fluids throughout the day. Wear insulated clothing. Buddy system shall be utilized. Adjust work schedule when needed.
General sampling activities	Vehicle traffic and equipment Exposure/contact with environmental media (e.g., contaminated soil, sediment, groundwater and waste materials). Sampling in/around excavation area.	Samplers shall be alert and know their surroundings at all times. Samplers shall don sampling gloves when sampling environmental media. Thorough hand wash/rinse shall be conducted after sampling activities. Please review "PPE/Safety Supplies" section. Please review and comply with AHA "Excavation Activities." **A Competent Person** shall be required during all sampling activities near/at the excavation. Samplers will **NOT** enter any excavation more than 5 feet bgs without following requirements of EM 385-1-1.

ACTIVITY HAZARD ANALYSIS

Activity: Visual Sweep Activities
Prepared by: Michael Garner
Reviewed by: Michael Dorman

Potential Hazards/Concerns	Control Measures
1. Fire	No smoking or flame producing material permitted during sweep operations. Provide fire extinguishers of appropriate size and number for on site use. Monitor fire danger and hazards provide information in daily site briefing. Employees practice due diligence to avoid creating fire hazard conditions. Practice good housekeeping.
2. Uneven walking working surfaces	Wear proper footwear for the work environment. Avoid hazardous walking and work surfaces, do not take unnecessary risks. Mark hazardous areas with fences, barriers, or demarcating tape to prevent inadvertent entry.
3. Unexploded Ordnance	Ensure only required personnel are in hazardous areas; avoid unnecessary personnel in the area. Avoid marked ordnance items Person will remain within the ordnance clear marked areas.
4. Heat stress	Maintain proper hydration Avoid alcohol use. Get proper rest. Take breaks as needed to prevent overheating. Where appropriate work clothes to avoid overheating.
5. Biological Hazards	Know the potential Biological Hazardous of the area, as reference in the SSHP Practice avoidance to prevent exposure. Use repellents if appropriate and as needed. Where appropriate PPE, such as Snake Chaps, Gloves, high-topped leather work shoes. Inform supervision of Biological Hazard sightings.
6. Remote Work	Ensure field communication is available and working. Use the buddy system. Ensure supervision is aware of your work location. Wear proper PPE. Use high visibility clothing.

ACTIVITY HAZARD ANALYSIS

Activity: UXO/OE Intrusive Operations Using Manual Tools and Methods
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• UXO/OE Intrusive Operations Using Manual Tools and Methods NOTE: These operations are only to be performed by USACE-qualified UXO Technicians and are not to be used by anyone else.	• UXO/OE	• Ensure Exclusion Zones are established to authorized Minimum Separation Distances for Non-Project Personnel and Team Separation Distances for Project Personnel. • Use the minimum number of personnel (not less than two) to conduct the operation and minimize their exposure time to UXO/OE. • Ensure all personnel are USACE-qualified UXO Technicians. • Do not conduct operations within 200 ft or Inhabited Building Distance for blast protection (whichever is greater) of other UXO/OE intrusive operations. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where operations will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • Observe general UXO/OE hazards and precautions. • Ensure compliance with the UXO/OE Work Plan and relevant SOPs.

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• UXO/OE Intrusive Operations Using Manual Tools and Methods (Contd.)	• Loud noise	• Reduce the volume level of detection equipment used for anomaly reacquisition before donning headset and engaging power.
	• Slips, trips and falls	• Wear work boots with lug soles. • Maintain awareness of hazards associated with uneven or wet terrain.
	• Dangerous animals and insects	• Maintain awareness of hazards associated with dangerous animals and insects. • Observe task PPE requirements.
	• Repetitive stress injury	• Shift detection equipment from one arm to the other when fatigued. • Take breaks when necessary.
	• Back injury	• Observe proper lifting techniques setting up or putting away equipment.
	• Thermal Stress	• Review Heat and Cold Stress AHAs.

ACTIVITY HAZARD ANALYSIS

Activity: UXO/OE Intrusive Operations Using Manual Tools and Methods (cont'd)
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PPE/SAFETY SUPPLIES	SAFETY INSPECTIONS	TRAINING

• Initial PPE will be modified Level D.
• UXO/OE Detector and related navigation and/or data-recording equipment
• Support Zone
  • Cell phone or Radio communication
  • Eyewash station
  • Fire extinguishers
  • First Aid kit
  • Drinking water
  • 911 Air horn
  • Spill containment supplies
  • Air monitoring equipment
  • Emergency decontamination supplies
	• Prior to use, ensure equipment is operational, calibrated according to operating manuals, and performing in accordance with required standards.
	• General Site Safety (Daily)
		• Ensure non-UXO Technician UXO/OE detection personnel observe general UXO/OE hazards and understand the requirement to be escorted by qualified UXO Technicians when they are within an exclusion zone.
		• Ensure UXO/OE detection personnel are qualified on the specific UXO/OE detection equipment to be used.
		• UXO/OE Detection Equipment Refresher.
		• General and local UXO/OE hazards and precautions.
		• Daily Tailgate Safety Meeting.
		• Review "UXO/OE Detection" AHA.
		• HAZWOPER Certification (40-hr, 8-hr).
		• OSHA Supervisory (Required for Managers and Supervisors).
		• First Aid and CPR (At least two employees).
		• SSHP review and sign-off.

ACTIVITY HAZARD ANALYSIS

Activity: UXO/OE Avoidance
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• UXO/OE Avoidance NOTE: These operations require two USACE qualified UXO Technicians, one of whom must be at least a UXO Technician Level II and be assigned as Team Leader.	• UXO/OE	• Do not approach within 200 ft or Inhabited Building Blast Protection Distance (whichever is greater) of intrusive UXO/OE operations. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where operations will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • UXO Technicians shall conduct a detector-aided surface access survey and a subsurface survey for anomalies before any type of activities commence, including foot and/or vehicular traffic. • Observe general UXO/OE hazards and precautions. • When UXO/OE is encountered, it will be avoided, marked and reported using an MMR Incident Report Form from the MMR Munitions Management Plan and other local protocols. Non UXO qualified personnel will be advised of its location and associated hazards.
PRINCIPLE STEPS	**POTENTIAL HAZARDS**	**RECOMMENDED CONTROLS**
• UXO/OE Avoidance (Contd.)	• Non UXO qualified personnel	• When escorting non UXO qualified personnel, UXO Technicians shall meet the party to be escorted, ascertain their specific requirements and objectives, and conduct a general work and safety briefing prior to commencing a transit of an area with potential UXO contamination. • Ensure all non UXO qualified personnel are thoroughly briefed about UXO/OE safety hazards and precautions anticipated at the project site. • Non UXO qualified personnel will follow behind UXO qualified escorts. • Team leaders and other assigned UXO Technicians will closely observe the whereabouts and behavior of non UXO qualified personnel to reduce their potential for exposure to UXO/OE.
	• Loud noise	• Reduce the volume level of detection equipment used for anomaly reacquisition before donning headset and engaging power.
	• Slips, trips and falls	• Wear work boots with lug soles. • Maintain awareness of hazards associated with uneven or wet terrain.
	• Dangerous animals and insects	• Maintain awareness of hazards associated with dangerous animals and insects. • Observe task PPE requirements.
	• Thermal Stress	• Review Heat and Cold Stress AHAs.

ACTIVITY HAZARD ANALYSIS

Activity: UXO/OE Detection
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• UXO/OE Detection NOTE: These operations are only to be performed under the supervision of USACE-qualified UXO Technicians and UXO/OE detection equipment operators.	• UXO/OE	• Do not approach within 200 ft or Inhabited Building Blast Protection Distance (whichever is greater) of intrusive UXO/OE operations. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where operations will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • UXO Technicians shall conduct a detector-aided surface access survey and a subsurface survey for anomalies before any type of activities commence, including foot and/or vehicular traffic. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where operations will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • UXO Technicians shall conduct a detector-aided surface access survey and a subsurface survey for anomalies before any type of activities commence, including foot and vehicular traffic. • Observe general UXO/OE hazards and precautions.
	• Loud noise	• Reduce the volume level of detection equipment before donning headset and engaging power.
	• Slips, trips and falls	• Wear work boots with lug soles. • Maintain awareness of hazards associated with uneven or wet terrain.
	• Dangerous animals and insects	• Maintain awareness of hazards associated with dangerous animals and insects. • Observe task PPE requirements.
	• Repetitive stress injury	• Shift detection equipment from one arm to the other when fatigued. • Take breaks when necessary.
	• Back injury	• Observe proper lifting techniques setting up or putting away equipment.
	• Thermal Stress	• Review Heat and Cold Stress AHAs.

ACTIVITY HAZARD ANALYSIS

Activity: UXO/OE Down-Hole Monitoring Support
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• UXO/OE Down-Hole Monitoring Support NOTE: These operations are only to be performed under the supervision of USACE-qualified UXO Technicians. NOTE: An access and site survey must precede any type of HTRW-related investigation activities or ingress/egress within an area identified or suspected of being contaminated with UXO/OE. At MMR, these surveys are also required to support "well pad" or "down-hole" monitoring activities.	• UXO/OE	• Do not approach within 200 ft or Inhabited Building Blast Protection Distance (whichever is greater) of intrusive UXO/OE operations. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where down-hole monitoring will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • For all down-hole monitoring activities (various depths), UXO Technicians shall visually inspect the down-hole monitoring site for UXO/OE. In addition, they will conduct a detector-aided search to ensure no UXO/OE is located below the surface of the area to be monitored to the depth necessary to facilitate specific monitoring requirements. • Observe general UXO/OE hazards and precautions. • When UXO/OE is encountered, it will be avoided, marked and reported using an MMR Incident Report Form from the MMR Munitions Management Plan and other local protocols. Non UXO qualified personnel will be advised of its location and associated hazards.
PRINCIPLE STEPS	**POTENTIAL HAZARDS**	**RECOMMENDED CONTROLS**
• UXO/OE Down-Hole Monitoring Support (Contd.)	• Non UXO qualified personnel	• When escorting non UXO qualified personnel, UXO Technicians shall meet the party to be escorted, ascertain their specific requirements and objectives, and conduct a general work and safety briefing prior to commencing a transit of an area with potential UXO contamination. • Ensure all non UXO qualified personnel are thoroughly briefed about UXO/OE safety hazards and precautions anticipated at the project site. • Non UXO qualified personnel will follow behind UXO qualified escorts. • Team leaders and other assigned UXO Technicians will closely observe the whereabouts and behavior of non UXO qualified personnel to reduce their potential for exposure to UXO/OE.
	• Loud noise	• Reduce the volume level of detection equipment used for anomaly reacquisition before donning headset and engaging power. • Wear adequate hearing protection when conducting drilling and monitoring equipment.
	• Slips, trips and falls	• Wear work boots with lug soles. • Maintain awareness of hazards associated with uneven or wet terrain.

ACTIVITY HAZARD ANALYSIS

Activity: Geophysical Detection, Surveying and Mapping
Prepared by: Michael Garner
Reviewed by: Michael Dorman

PRINCIPLE STEPS	POTENTIAL HAZARDS	RECOMMENDED CONTROLS
• Geophysical Detection, Surveying, and Mapping NOTE: These operations are only to be performed under the supervision of USACE-qualified UXO Technicians and operators qualified to use required equipment.	• UXO/OE	• Do not approach within 200 ft or Inhabited Building Blast Protection Distance (whichever is greater) of intrusive UXO/OE operations. • UXO Technicians shall comply with all requirements of USACE EP 75-1-2. • UXO Technicians shall review any archival information available regarding the area of MMR where operations will be conducted, and determine the level of risk associated with traversing the area and any additional specific safety considerations. • Observe general UXO/OE hazards and precautions.
	• Loud noise	• Reduce the volume level of detection equipment before donning headset and engaging power.
	• Slips, trips and falls	• Wear work boots with lug soles. • Maintain awareness of hazards associated with uneven or wet terrain.
	• Dangerous animals and insects	• Maintain awareness of hazards associated with dangerous animals and insects. • Observe task PPE requirements.
	• Repetitive stress injury	• Shift detection equipment from one arm to the other when fatigued. • Take breaks when necessary.
	• Back injury	• Observe proper lifting techniques setting up or putting away equipment.
	• Thermal Stress	• Review Heat and Cold Stress SOPs.

PPE/SAFETY SUPPLIES	SAFETY INSPECTIONS	TRAINING

• Initial PPE will be modified Level D.
• UXO/OE Detector and related navigation and/or data-recording equipment.
• <u>Support Zone</u>
  • Cell phone or Radio communication
  • Eyewash station
  • Fire extinguishers
  • First Aid kit
  • Drinking water
  • 911 Air horn
  • Spill containment supplies
  • Air monitoring equipment
  • Emergency decontamination supplies
	• Prior to use, ensure equipment is operational, calibrated according to operating manuals, and performing in accordance with required standards.
	• General Site Safety (Daily)
		• Ensure non-UXO Technician UXO/OE detection personnel observe general UXO/OE hazards and understand the requirement to be escorted by qualified UXO Technicians when they are within an exclusion zone.
		• Daily Tailgate Safety Meeting.
		• Review "UXO/OE Detection" AHA.
		• Ensure geophysical detection, survey and mapping personnel are qualified on the specific UXO/OE detection equipment to be used.
		• Required Equipment Refresher.
		• General and local UXO/OE hazards and precautions.
		• HAZWOPER Certification (40-hr, 8-hr).
		• OSHA Supervisory (Required for Managers and Supervisors).
		• First Aid and CPR (At least two employees).
		• SSHP review and sign-off.

Attachment 4
Emergency Contacts and Emergency Medical Care Locations

EMERGENCY PHONE NUMBERS

Life Threatening Emergencies: 911

Savanna Family Care Clinic
333 Chicago Avenue
Savanna, IL 61074
(815) 273-7110

Non-Life Threatening Emergencies:

Savanna Family Care Clinic
333 Chicago Avenue
Savanna, IL 61074
(815) 273-7110

Ambulance and Fire Department: 911

Poison Control Center: (800) 642-9999

Louisville District, U.S. Army Corps of Engineers Contacts:

Karen Rabek – Project Scientist
(502) 315-6328

Matthew Burg – USACE Safety and Occupational Health Manager
(502) 315-7061

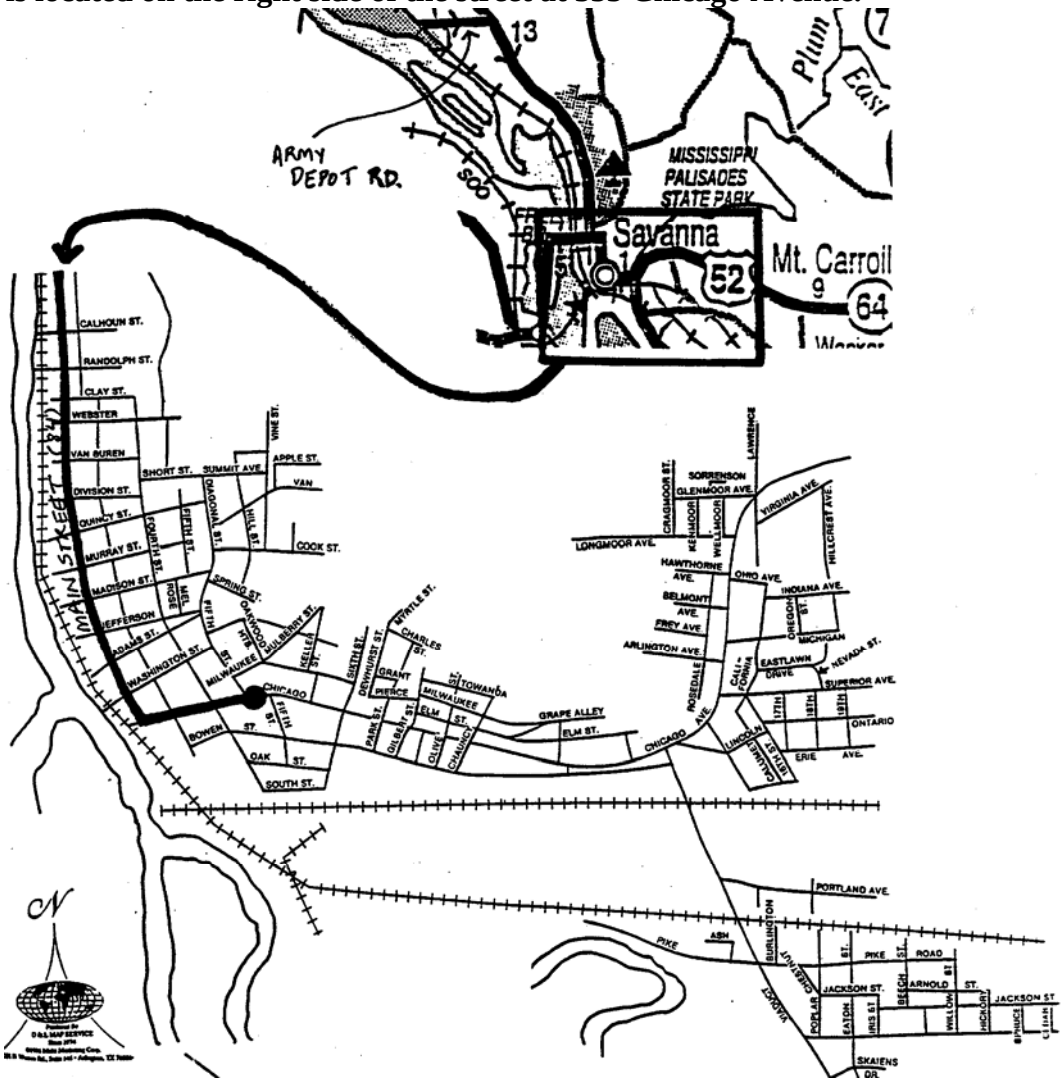
Client Contact:

John Clarke – Base Environmental Coordinator, Savanna Army Depot Activity
(815) 273-8827

Directions to the Savanna Family Care Clinic:

Savanna Family Care Clinic
333 Chicago Avenue
Savanna, IL 61074
(815) 273-7110

Exit Savanna Army depot through the main gate on Army Depot Road. From Army Depot Road, turn right onto Route 84 South (towards the town of Savanna). Once in Savanna, Route 84 is called Main Street. Continue along Main Street. Main Street veers to the left after the intersection with Washington Street. The Street becomes Chicago Avenue. The clinic is located on the right side of the street at 333 Chicago Avenue.



Attachment 5
ERT Incident and Injury Reporting Form

Earth Resources Technology, Inc.
Incident/Injury Report Form

Employee Information (to be completed by employee)

Name: _____ Work Phone: _____
Address: _____ Home Phone: _____
_____ Employee Number: _____
Date of Birth: _____ Male/Female: _____ Title: _____
Location of Work: _____ Part Time/Full Time: _____
Employment Dates: _____ Hours per day worked: _____
Days per week worked: _____ Regular Days off: _____

Accident Information (to be completed by employee)

Date of injury/illness: _____ Time of injury/illness: _____
Location (building/room) where injury/illness occurred: _____
What was employee doing when injured? How did injury occur?
_____
_____
_____
_____
_____
Describe the injury/illness (indicate type of injury, specify left or right, e.g. upper right leg):
_____
_____
Who was the incident reported to (Name/Title)? _____
Date Reported: _____ Time Reported: _____

Witness Names	Contact Information
_____	_____
_____	_____
_____	_____

Supervisor's Statement

Was Employee paid for full day? _____ Is Employee losing time? _____
Employee's first day away from work? _____ Has Employee returned? _____
Expected date of return: _____ Will employee be paid for lost time: _____
Did the employee receive medical attention? _____ Date Treated: _____
Name/Address of Medical Center: _____
_____
Who investigated the incident? _____ Title: _____
Telephone Number: _____ Email: _____

I CERTIFY THAT THE INFORMATION GIVEN ABOVE IS TRUE TO MY KNOWLEDGE

Employee Signature: _____ Date: _____

Supervisor Signature: _____ Date: _____

Attachment 6
USACE Engineering Form 3394

(For Safety Staff only)	REPORT NO.	EROC CODE **GO**	**UNITED STATES ARMY CORPS OF ENGINEERS** **ACCIDENT INVESTIGATION REPORT** *(For Use of this Form See Attached Instructions and USACE Suppl to AR 385-40)*	REQUIREMENT CONTROL SYMBOL: CEEC-S-8(R2)

1. ACCIDENT CLASSIFICATION

PERSONNEL CLASSIFICATION	INJURY/ILLNESS/FATAL	PROPERTY DAMAGE	MOTOR VEHICLE INVOLVED	DIVING
GOVERNMENT ☐ CIVILIAN ☐ MILITARY	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ CONTRACTOR	☐	☐ FIRE INVOLVED ☐ **OTHER**	☐	☐
☐ PUBLIC	☐ FATAL ☐ OTHER		☐	

2. PERSONAL DATA

a. NAME (Last, First, MI)	b. AGE	c. SEX ☐ MALE ☐ FEMALE	d. SOCIAL SECURITY NUMBER	e. GRADE

f. JOB SERIES/TITLE	g. DUTY STATUS	h. EMPLOYMENT STATUS AT TIME OF ACCIDENT
	☐ ON DUTY ☐ TDY ☐ OFF DUTY	☐ ARMY ACTIVE ☐ ARMY RESERVE ☐ VOLUNTEER ☐ PERMANENT ☐ FOREIGN NATIONAL ☐ SEASONAL ☐ TEMPORARY ☐ STUDENT ☐ OTHER (Specify)

3. GENERAL INFORMATION

a. DATE OF ACCIDENT *(month/day/year)*	b. TIME OF ACCIDENT *(military time)* hrs	c. EXACT LOCATION OF ACCIDENT	d. CONTRACTOR'S NAME (1) PRIME: (2) SUBCONTRACTOR

e. CONTRACT NUMBER	f. TYPE OF CONTRACT	g. HAZARDOUS/TOXIC WASTE
☐ CIVIL WORKS ☐ MILITARY ☐ OTHER (SPECIFY)	☐ CONSTRUCTION ☐ SERVICE ☐ A/E ☐ DREDGE ☐ OTHER (SPECIFY)_____	☐ SUPERFUND ☐ DERP ☐ IRP ☐ OTHER (SPECIFY) _____

4. CONSTRUCTION ACTIVITIES ***(Fill in line and corresponding code number in box from list - see instructions)***

a. CONSTRUCTION ACTIVITY	(CODE)	b. TYPE OF CONSTRUCTION EQUIPMENT	(CODE)
	#		#

5. INJURY/ILLNESS INFORMATION ***(Include name on line and corresponding code number in box for items e, f & g - see instructions)***

a. SEVERITY OF ILLNESS/INJURY	(CODE)	b. ESTIMATED DAYS LOST	c. ESTIMATED DAYS HOSPITALIZED	d. ESTIMATED DAYS REST. DUTY
	#			

e. BODY PART AFFECTED	(CODE)	g. TYPE AND SOURCE OF INJURY/ILLNESS	(CODE)
PRIMARY	#		
_____	(CODE) #	**TYPE**	(CODE) #
f. NATURE OF ILLNESS/INJURY	(CODE)	_____	(CODE)
	#		#

6. PUBLIC FATALITY ***(Fill in line and correspondence code number in box - see instructions)***

a. ACTIVITY AT TIME OF ACCIDENT	(CODE)	b. PERSONAL FLOATATION DEVICE USED?
	#	☐ YES ☐ NO ☐ N/A

7. MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE	b. TYPE OF COLLISION	c. SEAT BELTS	USED	NOT USED	NOT AVAILABLE
☐ PICKUP/VAN ☐ AUTOMOBILE ☐ TRUCK ☐ OTHER (Specify)	☐ SIDE SWIPE ☐ HEAD ON ☐ REAR END ☐ BROADSIDE ☐ ROLL OVER ☐ BACKING ☐ OTHER (Specify)	(1) FRONT SEAT (2) REAR SEAT			

8. PROPERTY/MATERIAL INVOLVED

a. NAME OF ITEM	b. OWNERSHIP	c. $ AMOUNT OF DAMAGE
(1)		
(2)		
(3)		

9. VESSEL/FLOATING PLANT ACCIDENT ***(Fill in line and correspondence code number in box from list - see instructions)***

a. TYPE OF VESSEL/FLOATING PLANT	(CODE)	b. TYPE OF COLLISION/MISHAP	(CODE)
	#		#

10. ACCIDENT DESCRIPTION (Use Additional paper, if necessary)

See attached page.

11. CASUAL FACTORS *(Read Instructions Before Completing)*

a. (Explain YES answers in item 13	YES	NO
DESIGN: Was design of facility, workplace or equipment a factor?	☐	☐
INSPECTION/MAINTENANCE: Were inspection & maintenance procedures a factor?	☐	☐
PERSON'S PHYSICAL CONDITION: In your opinion, was the physical condition of the person a factor?	☐	☐
OPERATING PROCEDURES: Were operating procedures a factor?	☐	☐
JOB PRACTICES: Were any job safety/health practices not followed when the accident occurred?	☐	☐
HUIMAN FACTORS: Did any human factors such as size or strength of person, etc., contribute to accident?	☐	☐
ENVIRONMENTAL FACTORS: Did heat, cold, dust, sun, glare, etc. contribute to the accident?	☐	☐

a. (CONTINUED)	YES	NO
CHEMICAL AND PHYSICAL AGENT FACTORS: Did exposure to chemical agents, such as dust, fumes, mists, vapors, or physical agents such as noice, radiation, etc. contribute to accident?	☐	☐
OFFICE FACTORS: Did office setting such as, lifting office furniture, carrying, stooping, etc. contribute to the accident?	☐	☐
SUPPORT FACTORS: Were inappropriate tools/resources provided to properly perform the activity/task?	☐	☐
PERSONAL PROTECTIVE EQPT: Did the improper selection, use or maintenance of personal protective eqpt contribute to the accident?	☐	☐
DRUGS/ALCOHOL: In your opinion, was deugs or alcohol factor to the accident?	☐	☐

b. WAS A WRITTEN JOB/ACTIVITY HAZARD ANALYSIS COMPLETED FOR TASK BEING PERFORMED AT TIME OF ACCIDENT?

☐ YES (If yes, attach a copy) ☐ NO

12. TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK?	b. TYPE OF TRAINING	c. DATE OF MOST RECENT FORMAL TRAINING
☐ YES ☐ NO	☐ CLASSROOM ☐ ON JOB	Month/Day/Year

13. FULLY EXPLAIN WHAT ALLOWED OR CAUSED THE ACCIDENT: INCLUDE DIRECT AND INDIRECT CAUSES (See instruction for definition of direct and indirect causes.) (Use additional paper, if necessary)

a. DIRECT CAUSE

See attached page.

b. INDIRECT CAUSE(S)

See attached page.

14. ACTION(S) TAKEN, ANTICIPATED OR RECOMMENDED TO ELIMINATE CAUSE(S)

DESCRIBE FULLY:

See attached page.

15. DATES FOR ACTIONS IDENTIFIED IN BLOCK 14

a. BEGINNING (Month/Day/Year)	b. ANTICIPATED COMPLETION (Month/Day/Year)

c. SIGNATURE AND TITLE OF SUPERVISOR CORPS CONTRACTOR	d. DATE (Month/Day/Year)	e. ORGANIZATION IDENTIFIER (Div,Br,Sect)	f. OFFICE SYMBOL

16. MANAGEMENT REVIEW (1st)

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

17. MANAGEMENT REVIEW (2nd - Chief Operations, Construction, Engineering, etc.)

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

18. SAFETY AND OCCUPATIONAL HEALTH OFFICE REVIEW

a. ☐ CONCUR b. ☐ NON CONCUR c. ADDITIONAL ACTIONS/COMMENTS

SIGNATURE	TITLE	DATE

19. COMMAND APPROVAL

COMMENTS

COMMANDER SIGNATURE DATE

* U.S. GOVERNMENT PRINTING OFFICE: 1993-0-791-757

10. ACCIDENT DESCRIPTION (Continuation)

13a. DIRECT CAUSE (Continuation)

13b. INDIRECT CAUSES (Continuation)
14. ACTION(S) TAKEN, ANTICIPATED, OR RECOMMENDED TO ELIMINATE CAUSE(S) (Continuation)

GENERAL. Complete a separate report for each person who was *injured, caused,* or *contributed* to the accident (excluding uninjured personnel and witnesses). Use of this form for reporting USACE employee first-aid type injuries not submitted to the Office of Workers' Compensation Programs (OWCP) shall be at the descretion of the FOA commander. Please type or print legibly. Appropriate items shall be marked with an "X" in box(es). If additional space is needed, provide the information on a separate sheet and attach to the completed form. Ensure that these instructions are forwarded with the completed report to the designated management reviewers indicated in sections 16. and 17.

INSTRUCTIONS FOR SECTION 1— ACCIDENT CLASSIFICATION. *(Mark All Boxes That Are Applicable.)*

a. GOVERNMENT. Mark "CIVILIAN" box if accident involved government civilian employee; mark "MILITARY" box if accident involved U.S. military personnel.
   (1) INJURY/ILLNESS/FATALITY—Mark if accident resulted in any government civilian employee injury, illness, or fatality that requires the submission of OWCP Forms CA-1 (injury), CA-2 (illness), or CA-6 (fatality) to OWCP; mark if accident resulted in military personnel lost-time or fatal injury or illness.
   (2) PROPERTY DAMAGE—Mark the appropriate box if accident resulted in any damage of $1000 or more to government property (including motor vehicles).
   (3) VEHICLE INVOLVED—Mark if accident involved a motor vehicle, *regardless* of whether "INJURY/ILLNESS/FATALITY" or "PROPERTY DAMAGE" are marked.
   (4) DIVING ACTIVITY—Mark if the accident involved an in-house USACE diving activity.

b. CONTRACTOR.
   (1) INJURY/ILLNESS/FATALITY—Mark if accident resulted in any contractor lost-time injury/illness or fatality.
   (2) PROPERTY DAMAGE—Mark the appropriate box if accident resulted in any damage of $1000 or more to contractor property (including motor vehicles).
   (3) VEHICLE INVOLVED—Mark if accident involved a motor vehicle, *regardless* of whether "INJURY/ILLNESS/FATALITY" or "PROPERTY DAMAGE" are marked.
   (4) DIVING ACTIVITY—Mark if the accident involved a USACE Contractor diving activity.

c. PUBLIC.
   (1) INJURY/ILLNESS/FATALITY—Mark if accident resulted in public fatality or permanent total disability. (The "OTHER" box will be marked when requested by the FOA to report an unusual non-fatal public accident that could result in claims against the government or as otherwise directed by the FOA Commander).
   (2) VOID SPACE—Make no entry.
   (3) VEHICLE INVOLVED—Mark if accident resulted in a fatality to a member of the public and involved a motor vehicle, *regardless* of whether "INJURY/ILLNESS/FATALITY" is marked.
   (4) VOID SPACE—Make no entry.

INSTRUCTIONS FOR SECTION 2—PERSONAL DATA

a. NAME—(MANDATORY FOR GOVERNMENT ACCIDENTS. OPTIONAL AT THE DISCRETION OF THE FOA COMMANDER FOR CONTRACTOR AND PUBLIC ACCIDENTS). Enter last name, first name, middle initial of person involved.

b. AGE—Enter age.

c. SEX—Mark appropriate box.

d. SOCIAL SECURITY NUMBER—(FOR GOVERNMENT PERSONNEL ONLY) Enter the social security number (or other personal identification number if no social security number issued).

e. GRADE—(FOR GOVERNMENT PERSONNEL ONLY) Enter pay grade. Example: O-6; E-7; WG-8; WS-12; GS-11; etc.

f. JOB SERIES/TITLE—For *government civilian employees* enter the pay plan, full series number, and job title, e.g. GS-0810/Civil Engineer. For *military personnel* enter the primary military occupational specialty (PMOS), e.g., 15A30 or 11G50. For *contractor employees* enter the job title assigned to the injured person, e.g. carpenter, laborer, surveyor, etc.,

g. DUTY STATUS—Mark the appropriate box.
   (1) ON DUTY—Person was at duty station during duty hours or person was away from duty station during duty hours but on official business at time of the accident.
   (2) TDY - Person was on official business, away from the duty station and with travel orders at time of accident. Line-of-duty investigation required.
   (3) OFF DUTY - Person was not on official business at time of accident

h. EMPLOYMENT STATUS—(FOR GOVERNMENT PERSONNEL ONLY) Mark the most appropriate box. If "OTHER" is marked, specify the employment status of the person.

INSTRUCTION FOR SECTION 3—GENERAL INFORMATION

a. DATE OF ACCIDENT—Enter the month, day, and year of accident.

b. TIME OF ACCIDENT—Enter the local time of accident in military time. Example: 1430 hrs (not 2:30 p.m.).

c. EXACT LOCATION OF ACCIDENT—Enter facts needed to locate the accident scene. (installation/project name, building number, street, direction and distance from closest landmark, etc.,).

d. CONTRACTOR NAME
   (1) PRIME—Enter the exact name (title of firm) of the prime contractor.
   (2) SUBCONTRACTOR—Enter the name of any subcontractor involved in the accident.

e. CONTRACT NUMBER—Mark the appropriate box to identify if contract is civil works, military, or other: if "OTHER" is marked, specify contract appropriation on line provided. Enter complete contract number of prime contract, e.g., DACW 09-85-C-0100.

f. TYPE OF CONTRACT—Mark appropriate box. A/E means architect/engineer. If "OTHER" is marked, specify type of contract on line provided.

g. HAZARDOUS/TOXIC WASTE ACTIVITY (HTW)—Mark the box to identify the HTW activity being performed at the time of the accident. For Superfund, DERP, and Installation Restoration Program (IRP) HTW activities include accidents that occurred during inventory, predesign, design, and construction. For the purpose of accident reporting, DERP Formerly Used DoD Site (FUDS) activities and IRP activities will be treated separately. For Civil Works O&M HTW activities mark the "OTHER" box.

INSTRUCTIONS FOR SECTION 4—CONSTRUCTION ACTIVITIES

a. CONSTRUCTION ACTIVITY—Select the *most appropriate* construction activity being performed at time of accident from the list below. Enter the activity name and place the corresponding code number identified in the box.

CONSTRUCTION ACTIVITY LIST

1. MOBILIZATION
2. SITE PREPARATION
3. EXCAVATION/TRENCHING
4. GRADING (EARTHWORK)
5. PIPING/UTILITIES
6. FOUNDATION
7. FORMING
8. CONCRETE PLACEMENT
9. STEEL ERECTION
10. ROOFING
11. FRAMING
12. MASONRY
13. CARPENTRY
14. ELECTRICAL
15. SCAFFOLDING/ACCESS
16. MECHANICAL
17. PAINTING
18. EQUIPMENT/MAINTENANCE
19. TUNNELING
20. WAREHOUSING/STORAGE
21. PAVING
22. FENCING
23. SIGNING
24. LANDSCAPING/IRRIGATION
25. INSULATION
26. DEMOLITION

INSTRUCTIONS FOR SECTION 13 — CAUSES

a. DIRECT CAUSES — The direct cause is that single factor which most directly lead to the accident. See examples below.

b. INDIRECT CAUSES — Indirect causes are those factors which contributed to but did not directly initiate the occurrence of the accident.

Examples for section 13:

a. Employee was dismantling scaffold and fell 12 feet from unguarded opening.
Direct cause: failure to provide fall protection at elevation.
Indirect causes: failure to enforce USACE safety requirements; improper training/motivation of employee (possibility that employee was not knowledgeable of USACE fall protection requirements or was lax in his attitude towards safety); failure to ensure provision of positive fall protection whenever elevated; failure to address fall protection during scaffold dismantling in phase hazard analysis.

b. Private citizen had stopped his vehicle at intersection for red light when vehicle was struck in rear by USACE vehicle. (note USACE vehicle was in proper/safe working condition).
Direct cause: failure of USACE driver to maintain control of and stop USACE vehicle within safe distance.
Indirect cause: Failure of employee to pay attention to driving (defensive driving).

INSTRUCTIONS FOR SECTION 14 — ACTION TO ELIMINATE CAUSE(S)

DESCRIPTION — Fully describe all the actions taken, anticipated, and recommended to eliminate the cause(s) and prevent reoccurrence of similar accidents/illnesses. Continue on blank sheets of paper if necessary to fully explain and attach to the completed report form.

INSTRUCTIONS FOR SECTION 15 — DATES FOR ACTION

a. BEGIN DATE — Enter the date when the corrective action(s) identified in Section 14 will begin.

b. COMPLETE DATE — Enter the date when the corrective action(s) identified in Section 14 will be completed.

c. TITLE AND SIGNATURE — Enter the title and signature of supervisor completing the accident report. For a GOVERNMENT employee accident/illness the immediate supervisor will complete and sign the report. For PUBLIC accidents the USACE Project Manager/Area Engineer responsible for the USACE property where the accident happened shall complete and sign the report. For CONTRACTOR accidents the Contractor's project manager shall complete and sign the report and provide to the USACE supervisor responsible for oversight of that contractor activity. This USACE Supervisor shall also sign the report. Upon entering the information required in 15.d, 15.e and 15.f below, the responsible USACE supervisor shall forward the report for management review as indicated in Section 16.

d. DATE SIGNED — Enter the month, day, and year that the report was signed by the responsible supervisor.

e. ORGANIZATION NAME — For GOVERNMENT employee accidents enter the USACE organization name (Division, Branch, Section, etc.) of the injured employee. For PUBLIC accidents enter the USACE organization name for the person identified in block 15.c. For CONTRACTOR accidents enter the USACE organization name for the USACE office responsible for providing contract administration oversight.

f. OFFICE SYMBOL — Enter the latest complete USACE Office Symbol for the USACE organization identified in block 15.e.

INSTRUCTIONS FOR SECTION 16 — MANAGEMENT REVIEW (1st)

1ST REVIEW — Each USACE FOA shall determine who will provide 1st management review. The responsible USACE supervisor in section 15.c shall forward the completed report to the USACE office designated as the 1st Reviewer by the FOA. Upon receipt, the Chief of the Office shall review the completed report, mark the appropriate box, provide substantive comments, sign, date, and forward to the FOA Staff Chief (2nd review) for review and comment.

INSTRUCTIONS FOR SECTION 17 — MANAGEMENT REVIEW (2nd)

2ND REVIEW — The FOA Staff Chief (i.e., FOA Chief of Construction, Operations, Engineering, Planning, etc.) shall mark the appropriate box, review the completed report, provide substantive comments, sign, date, and return to the FOA Safety and Occupational Health Office.

INSTRUCTIONS FOR SECTION 18 — SAFETY AND OCCUPATIONAL HEALTH REVIEW

3RD REVIEW — The FOA Safety and Occupational Health Office shall review the completed report, mark the appropriate box, ensure that any inadequacies, discrepancies, etc, are rectified by the responsible supervisor and management reviewers, provide substantive comments, sign, date and forward to the FOA Commander for review, comment, and signature.

INSTRUCTION FOR SECTION 19 — COMMAND APPROVAL

4TH REVIEW — The FOA Commander shall (to include the person designated Acting Commander in his absence) review the completed report, comment if required, sign, date, and forward the report to the FOA Safety and Occupational Health Office. Signature authority shall not be delegated.

b. TYPE OF CONSTRUCTION EQUIPMENT—Select the equipment involved in the accident from the list below. Enter the name and place the corresponding code number identified in the box. If equipment is not included below, use code 24, "OTHER", and write in specific type of equipment.

CONSTRUCTION EQUIPMENT

1. GRADER
2. DRAGLINE
3. CRANE (ON VESSEL/BARGE)
4. CRANE (TRACKED)
5. CRANE (RUBBER TIRE)
6. CRANE (VEHICLE MOUNTED)
7. CRANE (TOWER)
8. SHOVEL
9. SCRAPER
10. PUMP TRUCK (CONCRETE)
11. TRUCK (CONCRETE/TRANSIT MIXER)
12. DUMP TRUCK (HIGHWAY)
13. DUMP TRUCK (OFF HIGHWAY)
14. TRUCK (OTHER)
15. FORKLIFT
16. BACKHOE
17. FRONT-END LOADER
18. PILE DRIVER
19. TRACTOR (UTILITY)
20. MANLIFT
21. DOZER
22. DRILL RIG
23. COMPACTOR/VIBRATORY ROLLER
24. OTHER

INSTRUCTIONS FOR SECTION 5—INJURY/ILLNESS INFORMATION

a. SEVERITY OF INJURY / ILLNESS - Reference para 2-10 of USACE Suppl 1 to AR 385-40 and enter code and description from list below.

NOI NO INJURY
FAT FATALITY
PTL PERMANENT TOTAL DISABILITY
PPR PERMANENT PARTIAL DISABILITY
LWD LOST WORKDAY CASE INVOLVING DAYS AWAY FROM WORK
NLW RECORDABLE CASE WITHOUT LOST WORKDAYS
RFA RECORDABLE FIRST AID CASE
NRI NON-RECORDABLE INJURY

b. ESTIMATED DAYS LOST—Enter the estimated number of workdays the person will lose from work.

c. ESTIMATED DAYS HOSPITALIZED—Enter the estimated number of workdays the person will be hospitalized.

d. ESTIMATED DAYS RESTRICTED DUTY—Enter the estimated number of workdays the person, as a result of the accident, will not be able to perform all of their regular duties.

e. BODY PART AFFECTED—Select the most appropriate primary and when applicable, secondary body part affected from the list below. Enter body part name on line and place the corresponding code letters identifying that body part in the box.

GENERAL BODY AREA	CODE	BODY PART NAME
ARM/WRIST	AB	ARM AND WRIST
	AS	ARM OR WRIST
TRUNK, EXTERNAL MUSCULATURE	B1	SINGLE BREAST
	B2	BOTH BREASTS
	B3	SINGLE TESTICLE
	B4	BOTH TESTICLES
	BA	ABDOMEN
	BC	CHEST
	BL	LOWER BACK
	BP	PENIS
	BS	SIDE
	BU	UPPER BACK
	BW	WAIST
	BZ	TRUNK OTHER
HEAD, INTERNAL	C1	SINGLE EAR INTERNAL
	C2	BOTH EARS INTERNAL
	C3	SINGLE EYE INTERNAL
	C4	BOTH EYES INTERNAL
	CB	BRAIN
	CC	CRANIAL BONES
	CD	TEETH
	CJ	JAW
	CL	THROAT, LARYNX
	CM	MOUTH
	CN	NOSE
	CR	THROAT, OTHER
	CT	TONGUE
	CZ	HEAD OTHER INTERNAL
ELBOW	EB	BOTH ELBOWS
	ES	SINGLE ELBOW
FINGER	F1	FIRST FINGER
	F2	BOTH FIRST FINGERS
	F3	SECOND FINGER
	F4	BOTH SECOND FINGERS
	F5	THIRD FINGER
	F6	BOTH THIRD FINGERS
	F7	FOURTH FINGER
	F8	BOTH FOURTH FINGERS
TOE	G1	GREAT TOE
	G2	BOTH GREAT TOES
	G3	TOE OTHER
	G4	TOES OTHER
HEAD, EXTERNAL	H1	EYE EXTERNAL
	H2	BOTH EYES EXTERNAL
	H3	EAR EXTERNAL
	H4	BOTH EARS EXTERNAL
	HC	CHIN
	HF	FACE
	HK	NECK/THROAT
	HM	MOUTH/LIPS
	HN	NOSE
	HS	SCALP
KNEE	KB	BOTH KNEES
	KS	KNEE
LEG, HIP, ANKLE, BUTTOCK	LB	BOTH LEGS/HIPS/ ANKLES/BUTTOCKS
	LS	SINGLE LEG/HIP ANKLE/BUTTOCK
HAND	MB	BOTH HANDS
	MS	SINGLE HAND
FOOT	PB	BOTH FEET
	PS	SINGLE FOOT
TRUNK, BONES	R1	SINGLE COLLAR BONE
	R2	BOTH COLLAR BONES
	R3	SHOULDER BLADE
	R4	BOTH SHOULDER BLADES
	RB	RIB
	RS	STERNUM (BREAST BONE)
	RV	VERTEBRAE (SPINE; DISC)
	RZ	TRUNK BONES OTHER
SHOULDER	SB	BOTH SHOULDERS
	SS	SINGLE SHOULDER
THUMB	TB	BOTH THUMBS
	TS	SINGLE THUMB
TRUNK, INTERNAL ORGANS	V1	LUNG, SINGLE
	V2	LUNGS, BOTH
	V3	KIDNEY, SINGLE
	V4	KIDNEYS, BOTH
	VH	HEART
	VL	LIVER
	VR	REPRODUCTIVE ORGANS
	VS	STOMACH
	VV	INTESTINES
	VZ	TRUNK, INTERNAL; OTHER

f. NATURE OF INJURY/ILLNESS - Select the most appropriate nature of injury / illness from the list below. This nature of injury / illness shall correspond to the primary body part selected in 5e, above. Enter the nature of injury / illness name on the line and place the corresponding CODE letters in the box provided.

INSTRUCTIONS FOR SECTION 9 — VESSEL/ FLOATING PLANT ACCIDENT

a. TYPE OF VESSEL/FLOATING PLANT — Select the most appropriate vessel/floating plant from list below. Enter name and place corresponding number in box. If item is not listed below, enter item number for "OTHER" and write in specific type of vessel/ floating plant.

VESSEL/FLOATING PLANTS

1. ROW BOAT
2. SAIL BOAT
3. MOTOR BOAT
4. BARGE
5. DREDGE/HOPPER
6. DREDGE/SIDE CASTING
7. DREDGE/DIPPER
8. DREDGE/CLAMSHELL, BUCKET
9. DREDGE/PIPE LINE
10. DREDGE/DUST PAN
11. TUG BOAT
12. OTHER

b. COLLISION/MISHAP — Select from the list below the object(s) that contributed to the accident or were damaged in the accident.

COLLISION/MISHAP

1. COLLISION W/OTHER VESSEL
2. UPPER GUIDE WALL
3. UPPER LOCK GATES
4. LOCK WALL
5. LOWER LOCK GATES
6. LOWER GUIDE WALL
7. HAULAGE UNIT
8. BREAKING TOW
9. TOW BREAKING UP
10. SWEPT DOWN ON DAM
11. BUOY/DOLPHIN/CELL
12. WHARF OR DOCK
13. OTHER

INSTRUCTIONS FOR SECTION 10 — ACCIDENT DESCRIPTION

DESCRIBE ACCIDENT — Fully describe the accident. Give the sequence of events that describe what happened leading up to and including the accident. Fully identify personnel and equipment involved and their role(s) in the accident. Ensure that relationships between personnel and equipment are clearly specified. Continue on blank sheets if necessary and attach to this report.

INSTRUCTIONS FOR SECTION 11 — CAUSAL FACTORS

a. Review thoroughly. Answer each question by marking the appropriate block. If any answer is yes, explain in item 13 below. Consider, as a minimum, the following:

(1) DESIGN — Did inadequacies associated with the building or work site play a role? Would an improved design or layout of the equipment or facilities reduce the likelihood of similar accidents? Were the tools or other equipment designed and intended for the task at hand?

(2) INSPECTION/MAINTENANCE — Did inadequately or improperly maintained equipment, tools, workplace, etc. create or worsen any hazards that contributed to the accident? Would better equipment, facility, work site or work activity inspections have helped avoid the accident?

(3) PERSON'S PHYSICAL CONDITION — Do you feel that the accident would probably not have occurred if the employee was in "good" physical condition? If the person involved in the accident had been in better physical condition, would the accident have been less severe or avoided altogether? Was over exertion a factor?

(4) OPERATING PROCEDURES — Did a lack of or inadequacy within established operating procedures contribute to the accident? Did any aspect of the procedures introduce any hazard to, or increase the risk associated with the work process? Would establishment or improvement of operating procedures reduce the likelihood of similar accidents?

(5) JOB PRACTICES — Were any of the provisions of the Safety and Health Requirements Manual (EM 385-1-1) violated? Was the task being accomplished in a manner which was not in compliance with an established job hazard analysis or activity hazard analysis? Did any established job practice (including EM 385-1-1) fail to adequately address the task or work process? Would better job practices improve the safety of the task?

(6) HUMAN FACTORS — Was the person under undue stress (either internal or external to the job)? Did the task tend toward overloading the capabilities of the person; i.e., did the job require tracking and reacting to many external inputs such as displays, alarms, or signals? Did the arrangement of the workplace tend to interfere with efficient task performance? Did the task require reach, strength, endurance, agility, etc., at or beyond the capabilities of the employee? Was the work environment ill-adapted to the person? Did the person need more training, experience, or practice in doing the task? Was the person inadequately rested to perform safely?

(7) ENVIRONMENTAL FACTORS — Did any factors such as moisture, humidity, rain, snow, sleet, hail, ice, fog, cold, heat, sun, temperature changes, wind, tides, floods, currents, dust, mud, glare, pressure changes, lightning, etc., play a part in the accident?

(8) CHEMICAL AND PHYSICAL AGENT FACTORS — Did exposure to chemical agents (either single shift exposure or long-term exposure) such as dusts, fibers (asbestos, etc.), silica, gases (carbon monoxide, chlorine, etc.,), mists, steam, vapors, fumes, smoke, other particulates, liquid or dry chemicals that are corrosive, toxic, explosive or flammable, by-products of combustion or physical agents such as noise, ionizing radiation, non-ionizing radiation (UV radiation created during welding, etc.) contribute to the accident/incident?

(9) OFFICE FACTORS — Did the fact that the accident occurred in an office setting or to an office worker have a bearing on its cause? For example, office workers tend to have less experience and training in performing tasks such as lifting office furniture. Did physical hazards within the office environment contribute to the hazard?

(10) SUPPORT FACTORS — Was the person using an improper tool for the job? Was inadequate time available or utilized to safely accomplish the task? Were less than adequate personnel resources (in terms of employee skills, number of workers, and adequate supervision) available to get the job done properly? Was funding available, utilized, and adequate to provide proper tools, equipment, personnel, site preparation, etc?

(11) PERSONAL PROTECTIVE EQUIPMENT — Did the person fail to use appropriate personal protective equipment (gloves, eye protection, hard-toed shoes, respirator, etc.) for the task or environment? Did protective equipment provided or worn fail to provide adequate protection from the hazard(s)? Did lack of or inadequate maintenance of protective gear contribute to the accident?

(12) DRUGS/ALCOHOL — Is there any reason to believe the person's mental or physical capabilities, judgement, etc., were impaired or altered by the use of drugs or alcohol? Consider the effects of prescription medicine and over the counter medications as well as illicit drug use. Consider the effect of drug or alcohol induced "hangovers".

b. WRITTEN JOB/ACTIVITY HAZARD ANALYSIS — Was a written Job/Activity Hazard Analysis completed for the task being performed at the time of the accident? Mark the appropriate box. *If one was performed, attach a copy of the analysis to the report.*

INSTRUCTIONS FOR SECTION 12 — TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK? — For the purpose of this section "trained" means the person has been provided the necessary information (either formal and/or on-the-job (OJT) training) to competently perform the activity/task in a safe and healthful manner.

b. TYPE OF TRAINING — Mark the appropriate box that best indicates the type of training; (classroom or on-the-job) that the injured person received before the accident happened.

c. DATE OF MOST RECENT TRAINING — Enter the month, day, and year of the last *formal* training completed that covered the activity-task being performed at the time of the accident.

* The injury or condition selected below must be caused by a specific incident or event which occurred during a single work day or shift.

GENERAL NATURE CATEGORY	CODE	NATURE OF INJURY NAME
*TRAUMATIC INJURY OR DISABILITY	TA	AMPUTATION
	TB	BACK STRAIN
	TC	CONTUSION; BRUISE; ABRASION
	TD	DISLOCATION
	TF	FRACTURE
	TH	HERNIA
	TK	CONCUSSION
	TL	LACERATION, CUT
	TP	PUNCTURE
	TS	STRAIN, MULTIPLE
	TU	BURN, SCALD, SUNBURN
	TI	TRAUMATIC SKIN DISEASES/ CONDITIONS INCLUDING DERMATITIS
	TR	TRAUMATIC RESPIRATORY DISEASE
	TQ	TRAUMATIC FOOD POISONING
	TW	TRAUMATIC TUBERCULOSIS
	TX	TRAUMATIC VIROLOGICAL/ INFECTIVE/PARASITIC DISEASE
	T1	TRAUMATIC CEREBRAL VASCULAR CONDITION/STROKE
	T2	TRAUMATIC HEARING LOSS
	T3	TRAUMATIC HEART CONDITION
	T4	TRAUMATIC MENTAL DISORDER; STRESS; NERVOUS CONDITION
	T8	TRAUMATIC INJURY – OTHER (EXCEPT DISEASE, ILLNESS)

**A nontraumatic physiological harm or loss of capacity produced by systemic infection; continued or repeated stress or strain; exposure to toxins, poisons, fumes, etc.; or other continued and repeated exposures to conditions of the work environment over a long period of time. For practical purposes, an occupational illness/disease or disability is any reported condition which doses not meet the definition of traumatic injury or disability as described above.

GENERAL NATURE CATEGORY	CODE	NATURE OF INJURY NAME
**NON-TRAUMATIC ILLNESS/DISEASE OR DISABILITY		
RESPIRATORY DISEASE	RA	ASBESTOSIS
	RB	BRONCHITIS
	RE	EMPHYSEMA
	RP	PNEUMOCONIOSIS
	RS	SILICOSIS
	R9	RESPIRATORY DISEASE, OTHER
VIROLOGICAL, INFECTIVE & PARASITIC DISEASES	VB	BRUCELLOSIS
	VC	COCCIDIOMYCOSIS
	VF	FOOD POISONING
	VH	HEPATITIS
	VM	MALARIA
	VS	STAPHYLOCOCCUS
	VT	TUBERCULOSIS
	V9	VIROLOGICAL/INFECTIVE/ PARASITIC – OTHER
DISABILITY, OCCUPATIONAL	DA	ARTHRITIS, BURSITIS
	DB	BACK STRAIN, BACK SPRAIN
	DC	CEREBRAL VASCULAR CONDITION; STROKE
	DD	ENDEMIC DISEASE (OTHER THAN CODE TYPES R&S)
	DE	EFFECT OF ENVIRONMENTAL CONDITION
	DH	HEARING LOSS
	DK	HEART CONDITION
	DM	MENTAL DISORDER, EMOTIONAL STRESS NERVOUS CONDITION
	DR	RADIATION
	DS	STRAIN, MULTIPLE
	DU	ULCER
	DV	OTHER VASCULAR CONDITIONS
	D9	DISABILITY, OTHER
SKIN DISEASE OR CONDITION	SB	BIOLOGICAL
	SC	CHEMICAL
	S9	DERMATITIS, UNCLASSIFIED

g. TYPE AND SOURCE OF INJURY/ILLNESS (CAUSE) - Type and Source Codes are used to describe what caused the incident. The Type Code stands for an ACTION and the Source Code for an OBJECT or SUBSTANCE. Together, they form a brief description of how the incident occurred. Where there are two different sources, code the initiating source of the incident (see example 1, below). Examples:

(1) An employee tripped on carpet and struck his head on a desk.
TYPE: 210 (fell on same level) SOURCE: 0110 (walking/working surface)

NOTE: This example would NOT be coded 120 (struck against) and 0140 (furniture).

(2) A Park Ranger contracted dermatitis from contact with poison ivy/ oak.
TYPE: 510 (contact) SOURCE: 0920 (plant)

(3) A lock and dam mechanic punctured his finger with a metal sliver while grinding a turbine blade.
TYPE: 410 (punctured by) SOURCE: 0830 (metal)

(4) An employee was driving a government vehicle when it was struck by another vehicle..
TYPE: 800 (traveling in) SOURCE: 0421 (government-owned vehicle, as driver)

NOTE: The Type Code 800, "Traveling In" is different from the other type codes in that its function is not to identify factors contributing to the injury or fatality, but rather to collect data on the type of vehicle the employee was operating or traveling in at the time of the incident.

Select the most appropriate TYPE and SOURCE identifier from the list below and enter the name on the line and the corresponding code in the appropriate box.

CODE	TYPE OF INJURY NAME
	STRUCK
0110	STRUCK BY
0111	STRUCK BY FALLING OBJECT
0120	STRUCK AGAINST
	FELL, SLIPPED, TRIPPED
0210	FELL ON SAME LEVEL
0220	FELL ON DIFFERENT LEVEL
0230	SLIPPED, TRIPPED (NO FALL)
	CAUGHT
0310	CAUGHT ON
0320	CAUGHT IN
0330	CAUGHT BETWEEN
	PUNCTURED, LACERATED
0410	PUNCTURED BY
0420	CUT BY
0430	STUNG BY
0440	BITTEN BY
	CONTACTED
0510	CONTACTED WITH (INJURED PERSON MOVING)
0520	CONTACTED BY (OBJECT WAS MOVING)
	EXERTED
0610	LIFTED, STRAINED BY (SINGLE ACTION)
0620	STRESSED BY (REPEATED ACTION)
	EXPOSED
0710	INHALED
0720	INGESTED
0730	ABSORBED
0740	EXPOSED TO
0800	TRAVELING IN

CODE	SOURCE OF INJURY NAME
0100	BUILDING OR WORKING AREA
0110	WALKING/WORKING SURFACE (FLOOR, STREET, SIDEWALKS, ETC)
0120	STAIRS, STEPS
0130	LADDER
0140	FURNITURE, FURNISHINGS, OFFICE EQUIPMENT
0150	BOILER, PRESSURE VESSEL
0160	EQUIPMENT LAYOUT (ERGONOMIC)
0170	WINDOWS, DOORS
0180	ELECTRICITY

CODE	SOURCE OF INJURY NAME
0200	ENVIRONMENTAL CONDITION
0210	TEMPERATURE EXTREME (INDOOR)
0220	WEATHER (ICE, RAIN, HEAT, ETC.)
0230	FIRE, FLAME, SMOKE (NOT TOBACCO)
0240	NOISE
0250	RADIATION
0260	LIGHT
0270	VENTILATION
0271	TOBACCO SMOKE
0280	STRESS (EMOTIONAL)
0290	CONFINED SPACE
0300	MACHINE OR TOOL
0310	HAND TOOL (POWERED: SAW, GRINDER, ETC.)
0320	HAND TOOL (NONPOWERED)
0330	MECHANICAL POWER TRANSMISSION APPARATUS
0340	GUARD, SHIELD (FIXED, MOVEABLE, INTERLOCK)
0350	VIDEO DISPLAY TERMINAL
0360	PUMP, COMPRESSOR, AIR PRESSURE TOOL
0370	HEATING EQUIPMENT
0380	WELDING EQUIPMENT
0400	VEHICLE
0411	AS DRIVER OF PRIVATELY OWNED/RENTAL VEHICLE
0412	AS PASSENGER OF PRIVATELY OWNED/RENTAL VEHICLE
0421	DRIVER OF GOVERNMENT VEHICLE
0422	PASSENGER OF GOVERNMENT VEHICLE
0430	COMMON CARRIER (AIRLINE, BUS, ETC.)
0440	AIRCRAFT (NOT COMMERCIAL)
0450	BOAT, SHIP, BARGE
0500	MATERIAL HANDLING EQUIPMENT
0510	EARTHMOVER (TRACTOR, BACKHOE, ETC.)
0520	CONVEYOR (FOR MATERIAL AND EQUIPMENT)
0530	ELEVATOR, ESCALATOR, PERSONNEL HOIST
0540	HOIST, SLING CHAIN, JACK
0550	CRANE
0551	FORKLIFT
0560	HANDTRUCK, DOLLY
0600	DUST, VAPOR, ETC.
0610	DUST (SILICA, COAL, ETC.)
0620	FIBERS
0621	ASBESTOS
0630	GASES
0631	CARBON MONOXIDE
0640	MIST, STEAM, VAPOR, FUME
0641	WELDING FUMES
0650	PARTICLES (UNIDENTIFIED)
0700	CHEMICAL, PLASTIC, ETC.
0711	DRY CHEMICAL—CORROSIVE
0712	DRY CHEMICAL—TOXIC
0713	DRY CHEMICAL—EXPLOSIVE
0714	DRY CHEMICAL—FLAMMABLE
0721	LIQUID CHEMICAL—CORROSIVE
0722	LIQUID CHEMICAL—TOXIC
0723	LIQUID CHEMICAL—EXPLOSIVE
0724	LIQUID CHEMICAL—FLAMMABLE
0730	PLASTIC
0740	WATER
0750	MEDICINE
0800	INANIMATE OBJECT
0810	BOX, BARREL, ETC.
0820	PAPER
0830	METAL ITEM, MINERAL
0831	NEEDLE
0840	GLASS
0850	SCRAP, TRASH
0860	WOOD
0870	FOOD
0880	CLOTHING, APPAREL, SHOES
0900	ANIMATE OBJECT
0911	DOG
0912	OTHER ANIMAL
0920	PLANT
0930	INSECT
0940	HUMAN (VIOLENCE)
0950	HUMAN (COMMUNICABLE DISEASE)
0960	BACTERIA, VIRUS (NOT HUMAN CONTACT)

CODE	SOURCE OF INJURY NAME
1000	PERSONAL PROTECTIVE EQUIPMENT
1010	PROTECTIVE CLOTHING, SHOES, GLASSES, GOGGLES
1020	RESPIRATOR, MASK
1021	DIVING EQUIPMENT
1030	SAFETY BELT, HARNESS
1040	PARACHUTE

INSTRUCTIONS FOR SECTION 6 — PUBLIC FATALITY

a. ACTIVITY AT TIME OF ACCIDENT—Select the activity being performed at the time of the accident from the list below. Enter the activity name on the line and the corresponding number in the box. If the activity performed is not identified on the list, select from the *most* appropriate primary activity area (water related, non-water related or other activity), the code number for "Other", and write in the activity being performed at the time of the accident.

WATER RELATED RECREATION

1. Sailing
2. Boating—powered
3. Boating—unpowered
4. Water skiing
5. Fishing from boat
6. Fishing from bank dock or pier
7. Fishing while wading
8. Swimming/supervised area
9. Swimming/designated area
10. Swimming/other area
11. Underwater activities (skin diving, scuba, etc.)
12. Wading
13. Attempted rescue
14. Hunting from boat
15. Other

NON-WATER RELATED RECREATION

16. Hiking and walking
17. Climbing (general)
18. Camping/picnicking authorized area
19. Camping/picnicking unauthorized area
20. Guided tours
21. Hunting
22. Playground equipment
23. Sports/summer (baseball, football, etc.)
24. Sports/winter (skiing, sledding, snowmobiling etc.)
25. Cycling (bicycle, motorcycle, scooter)
26. Gliding
27. Parachuting
28. Other non-water related

OTHER ACTIVITIES

29. Unlawful acts (fights, riots, vandalism, etc.)
30. Food preparation/serving
31. Food consumption
32. Housekeeping
33. Sleeping
34. Pedestrian struck by vehicle
35. Pedestrian other acts
36. Suicide
37. "Other" activities

b. PERSONAL FLOTATION DEVICE USED—If fatality was water-related was the victim wearing a person flotation device? Mark the appropriate box.

INSTRUCTIONS FOR SECTION 7—MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE—Mark appropriate box for each vehicle involved. If more than one vehicle of the same type is involved, mark both halves of the appropriate box. USACE vehicle(s) involved shall be marked in left half of appropriate box.

b. TYPE OF COLLISION—Mark appropriate box.

c. SEAT BELT—Mark appropriate box.

INSTRUCTIONS FOR SECTION 8—PROPERTY/ MATERIAL INVOLVED.

a. NAME OF ITEM—Describe all property involved in accident. Property/material involved means material which is damaged or whose use or misuse contributed to the accident. Include the name, type, model; also include the National Stock Number (NSN) whenever applicable.

b. OWNERSHIP—Enter ownership for each item listed. (Enter one of the following: *USACE; OTHER GOVERNMENT; CONTRACTOR: PRIVATE)*

c. $ AMOUNT OF DAMAGE—Enter the total estimated dollar amount of damage (parts and labor), if any.

Attachment 7
OHSA Log 301 Form

OSHA's Form 301
Injuries and Illnesses Incident Report

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

This *Injury and Illness Incident Repor* t is one of the first forms you must fill out when a recordable work-related injury or illness has occurred. Together with the *Log of Work-Related injuries and Illnesses* and the accompanying *Summary*, these forms help the employer and OSHA develop a picture of the extent and severity of work-related incidents.

Within 7 calendar days after you receive information that a recordable work-related injury or illness has occurred, you must fill out this form or an equivalent. Some state workers' compensation, insurance, or other reports may be acceptable substitutes. To be considered an equivalent form, any substitute must contain all the information asked for on this form.

According to Public Law 91-596 and 29 CFR 1904, OSHA's recordkeeping rule, you must keep this form on file for 5 years following the year to which it pertains

If you need additional copies of this form, you may photocopy and use as many as you need.

Completed by _____

Title _____

Phone _____ Date _____

Information about the employee

1) Full Name _____

2) Street _____

City _____ State _____ Zip _____

3) Date of birth _____

4) Date hired _____

5) ☐ Male
☐ Female

Information about the physician or other health care professional

6) Name of physician or other health care professional

_____

_____

7) If treatment was given away from the worksite, where was it given?

Facility _____

Street _____

City _____ State _____ Zip _____

8) Was employee treated in an emergency room?
☐ Yes
☐ No

9) Was employee hospitalized overnight as an in-patient?
☐ Yes
☐ No

Information about the case

10) Case number from the Log _____ *(Transfer the case number from the Log after you record the case.)*

11) Date of injury or illness _____

12) Time employee began work _____ AM/PM

13) Time of event _____ AM/PM ☐ Check if time cannot be determined

14) **What was the employee doing just before the incident occurred?** Describe the activity, as well as the tools, equipment or material the employee was using. Be specific. Examples: "climbing a ladder while carrying roofing materials"; "spraying chlorine from hand sprayer"; "daily computer key-entry."

15) **What happened?** Tell us how the injury occurred. Examples: "When ladder slipped on wet floor, worker fell 20 feet"; "Worker was spayed with chlorine when gasket broke during replacement"; "Worker developed soreness in wrist over time."

16) **What was the injury or illness?** Tell us the part of the body that was affected and how it was affected; be more specific than "hurt", "pain", or "sore." Examples: "strained back"; "chemical burn, hand"; "carpal tunnel syndrome."

17) **What object or substance directly harmed the employee?** Examples: "concrete floor"; "chlorine"; "radial arm saw." If this question does not apply to the incident, leave it blank.

18) **If the employee died, when did death occur?** Date of death

Public reporting burden for this collection of information is estimated to average 22 minutes per response, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Persons are not required to respond to the collection of information unless it displays a current valid OMB control number. If you have any comments about this estimate or any other aspects of this data collection, including suggestions for reducing this burden, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave, NW, Washington, DC 20210. Do not send the completed forms to this office.

APPENDIX B
GEOPHYSICAL INVESTIGATION PLAN

FINAL
GEOPHYSICAL INVESTIGATION PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION AREA (SVAD-050),
SAVANNA ARMY DEPOT
SAVANNA, ILLINOIS

Prepared for:

U.S. Army Corps of Engineers
Louisville District

Contract W912QR-08-D-0012
Delivery Order 0001

Prepared by:

Earth Resources Technology, Inc.
4890 University Square, Suite 2A
Huntsville, Alabama 35816
(256) 217-8991

September 2009

FINAL
GEOPHYSICAL INVESTIGATION PLAN
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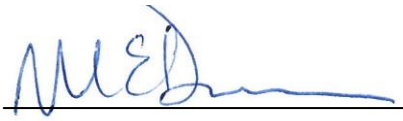
APPROVED BY



9/2/09

Michael Dorman
Project Manager

Date

5/20/09

Glenn Earhart
Program Manager

Date

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ACRONYMS AND ABBREVIATIONS

DID	Data Item Description
DQO	Data Quality Objective
EDM	Electronic Distance Measuring Device
EM	Electromagnetic
ERT	Earth Resources Technology, Inc.
FS	Feasibility Study
ft	feet
GIP	Geophysical Investigation Plan
GPO	Geophysical Prove-out
MEC	Munitions and Explosives of Concern
mph	Miles per Hour
OB	Open Burn
OBG	Old Burning Grounds
OD	Open Detonation
QA	Quality Assurance
QC	Quality Control
RI	Remedial Investigation
RTS	Robotic Total Station
SOW	Scope of Work
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
TDEM	Time Domain Electromagnetic
UXO	Unexploded Ordnance
USACE	United States Army Corps of Engineers

1.0 INTRODUCTION

Earth Resources Technology, Inc. (ERT) has been contracted by the U.S. Army Corps of Engineers (USACE), Louisville District to collect environmental and geophysical data to support Remedial Investigation (RI) report and Feasibility Study (FS) development for Savanna Army Depot Activity Site-50 (SVAD-050) of the Savanna Army Depot, in Savanna Illinois. This Geophysical Investigation Plan (GIP) was prepared to document the geophysical approach, methods, and operational procedures of the geophysical investigation component of RI data collection.

2.0 PROJECT OBJECTIVES

The overall project objective is to characterize the nature and extent of potential contamination at the SVDA Open Burn/Open Detonation (SVAD-050) site. One component of the project is to perform geophysical investigations at specified locations in order to locate and characterize anomalies that may represent potential Munitions and Explosives of Concern (MEC) burial pits and individual ordnance items. Data collected during this investigation will be used to prepare maps and dig sheets with recommendations for intrusive investigations. The results from geophysical and intrusive investigations will be included in a RI report.

Specific Data Quality Objectives (DQOs), related to the geophysical investigation, for this project are included in Section 6.2.

3.0 PROJECT ORGANIZATION

The technical team consists of USACE, Louisville District and ERT personnel. The roles of these team members are described below.

3.1 U.S. Army Corps of Engineers, Louisville District

USACE, Louisville District is the lead agency for this project. USACE's responsibilities include assignment of delivery orders to perform investigations, review of project plans and documents, working with the public, and coordinating with state and local regulatory agencies.

3.2 Earth Resource Technology, Inc.

ERT is responsible for performance of the activities detailed in the Work Plan and the Scope of Work (SOW). ERT is also responsible for schedule and budget control. ERT will provide overall engineering support and services for this project, including personnel to prepare work plans, perform investigation activities, evaluate results, and prepare reports.

Personnel performing work in support of the geophysical investigations will meet the qualifications required by Data Item Description (DID) MR-025.01. Figure 3-1 presents the geophysical investigation personnel organizational chart. The main Work Plan document lists specific personnel that have been tasked to this project.

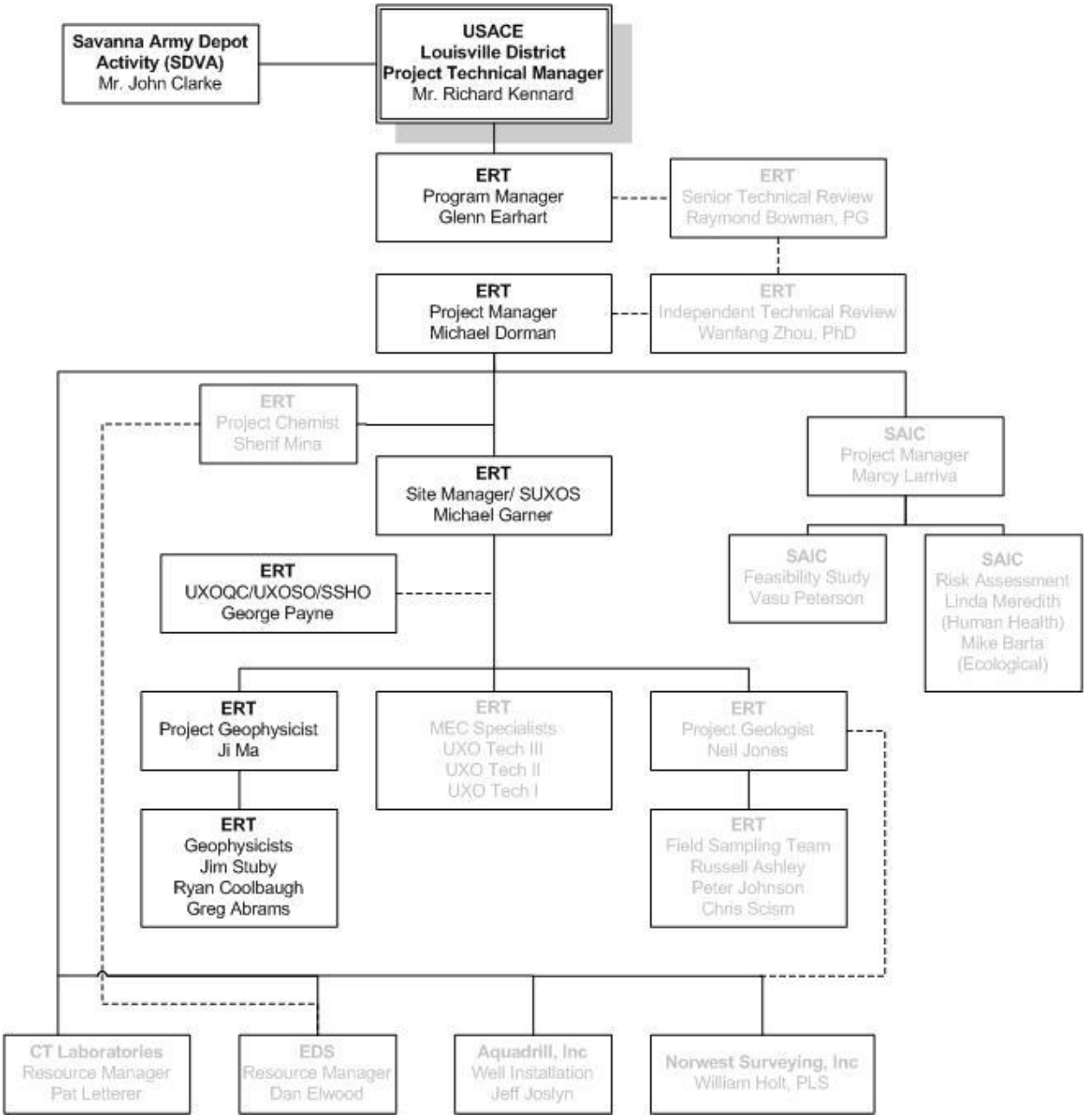


Figure 3-1. Geophysical Investigation Project Organization

4.0 GEOPHYSICAL PROVE-OUT PLAN

The project requires a site-specific Geophysical Prove-Out (GPO) for the purpose of evaluating geophysical instruments and developing the standard response for the selected instruments, instrument configuration, and techniques. The GPO will be performed in accordance with DID MR-005-05.01. Prior to geophysical investigations, a letter report summarizing the results of the GPO survey will be submitted for approval to USACE, Louisville District. Included in the letter report will be a standard dig sheet listing geophysical targets with X, Y coordinates and geophysical response at a minimum.

The specific DQO's for the GPO will be to:

- Demonstrate that the geophysical investigation systems/equipment are operating properly.
- Assess the operator's performance and update related procedures and assist in the development of operator measurement techniques.
- Establish a baseline of performance capabilities for the selected instruments.
- Establish decision parameters for target selection by the site geophysicists.
- Evaluate navigational/position systems for electronic positional accuracy for grid establishment and positioning of identified geophysical anomalies.
- Determine average speed, minimum along track sampling, line separation distance are required to detect all target items in the investigation areas.
- Determine appropriate latency corrections that account for instrument latency time and sensor velocity and evaluate the correction routine.
- Establish a data processing routine that does not alter the peak responses of anomalies by more than 10%.
- Ensure that data positioning errors in the final datasets will not exceed 20 centimeters.

4.1 GPO Test Plot

An existing GPO test plot is located north of the project site, as shown in Figure 4-1**.** This test plot was constructed by SAIC and was used in 2006 prior to geophysical investigations of the Old Burning Grounds (OBG). The OBG and SVAD-050 have been used similarly by the Army for munitions demolition, and the expected MEC to be located within SVAD-050 should be similar to items within the OBG. ERT will utilize the existing test plot to perform GPO investigations. Table 4-1 provides descriptions and locations of seeded items with the GPO test plot. Figure 4-2 is a map of the test plot with seed item locations shown. Additional blind seed items may have been placed at this test plot by the USACE.

4.2 Quality Control

Prior to conducting Quality Control (QC) function tests, an area is established as having "no noise" for use as a calibration base station around the proposed DGM survey site. Static background, static response, and vibration/cable connection tests are performed daily throughout the project, as well as on days when data is collected for the GPO. Quality control tests as outlined in Table 4-2 will be followed. A brief description of each test is included below. In

addition to these tests, repeat data will be collected over 3% of the GPO test plot, and over 3% of each production grid.

4.2.1 Static Background Test

When the instrument has been powered up sufficiently long to warm the electronics, the instrument will be placed on a non-metal stand to keep it stationary during the test. Readings will be collected for a minimum of three minutes after instrument warm-up. Data collected during static tests will be retained for documentation purposes. The effects of ambient noise may vary across a project site. Therefore, it may be necessary to perform several static tests across the survey area. The purpose of performing a static test is to determine whether a particular geophysical instrument is collecting stable readings. Improper instrument function, the presence of local sources of ambient noise (such as EM transmissions from high-voltage electric lines), and instability in the earth's magnetic field (as during a magnetic storm) are all potential causes of inconsistent, non-repeatable readings. The operator must review the readings to confirm their stability prior to continuing with the geophysical survey.

4.2.2 Static Response (Spike) Test

The instrument response test quantifies the response of the instrument to a standard test item. A standard 2" diameter trailer ball with integrated shaft should be used as the test item. Leaving the instrument in the same position as used in the Static Background Test, the test item, or spike, is placed below the sensor, then data is collected for a minimum three minute period. The test will document the amplitude of response to the test item and instrument drift.

4.2.3 Personnel Test

Test each person who will be on-site and near the equipment to ensure they are "metallic free" by having each person approach the detector while it is in a stationary mode and observing any deviations from background readings.

4.2.4 Six Line Test

This test is performed only on the first day of the project, at the GPO site. The test will be used to evaluate heading effects, repeatability of response amplitude, positional accuracy, and lantency. Six lines of data are collected along the same 50-foot long transect. For the first two passes over the transect there is no test item, or spike, and the operator moves at a normal pace down the transect then back in the opposite direction. For the second two passes a spike is placed on the ground at the halfway point, and the operator moves down and back along the transect at a normal pace again. For the fifth line the operator moves at a slower than normal pace along the transect with the spike left in place, and for the sixth line the operator moves at a faster than normal pace with the spike left in place. Additional, daily latency tests may be collected during production work, should the Project Geophysicist deem them necessary.

4.3 GPO Letter Report

The results of the GPO will be submitted in the GPO letter report in tabular and graphical form. The GPO Letter report will include, at a minimum, the following information:

- As-built drawing of the GPO plot;
- Color-coded maps of the geophysical data;
- Dig sheet;

- Summary of the GPO results; and
- Proposed geophysical equipment, techniques, and methodologies.

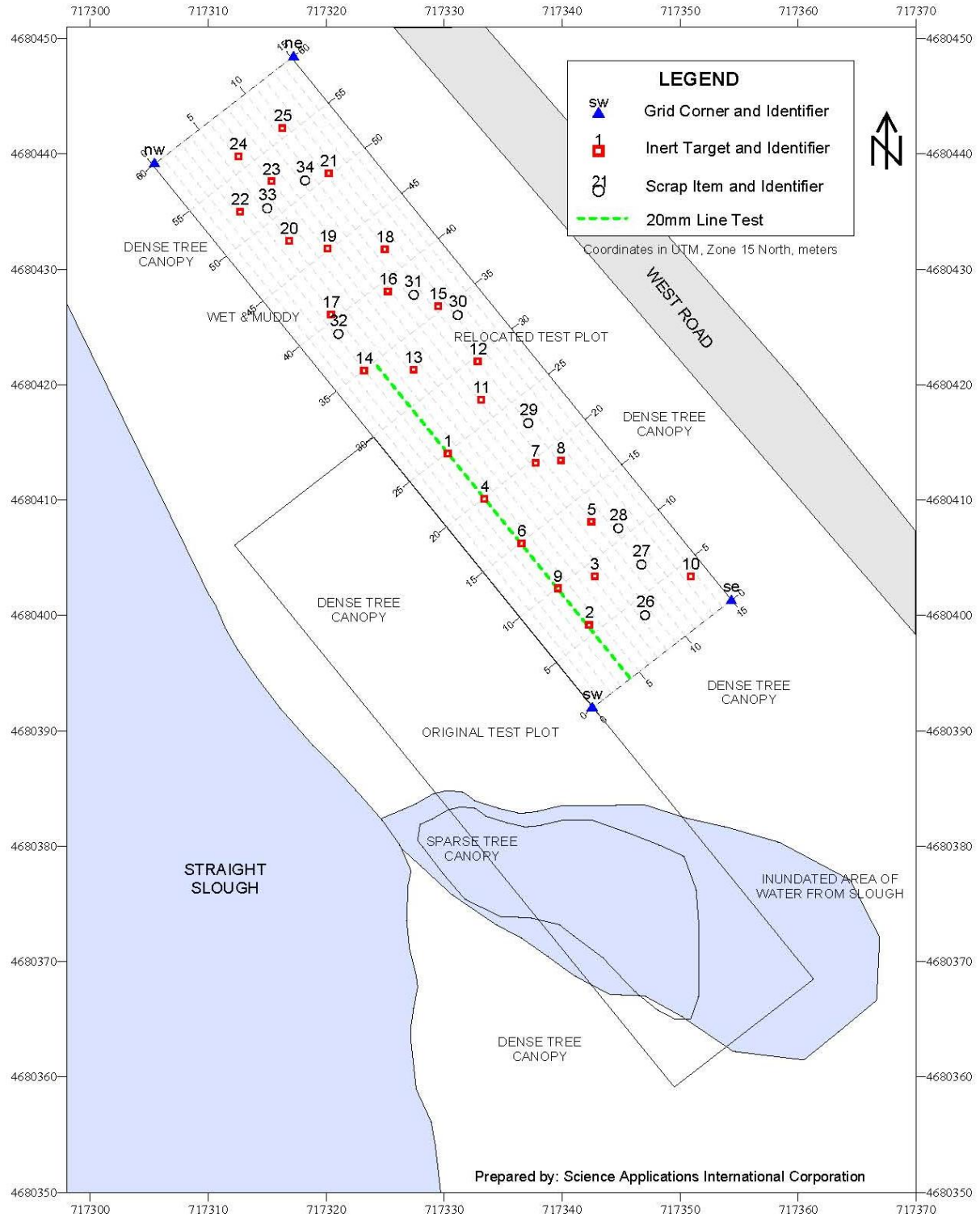
In addition, a CD shall be delivered with the letter report containing the following files:

- The GPO Letter Report (Adobe pdf);
- All raw and processed geophysical data. All data, except raw instrument data, shall be provided in column delineated ASCII files in the format x, y, z, v1, v2, etc., where x and y are coordinates (or equivalent) in Easting (feet [ft]) and Northing (ft) directions, z (elevation) is an optional field in ft, and v1, v2, v3, etc., are the instrument readings. A time stamp for the measurements will also be included. Each data field will be separated by a comma or tab for the measurements;
- Geophysical maps in their native format (Geosoft Oasis Montaj™ format) and/or as raster bit-map images such as BMP, JPEG, TIFF or PDF; and
- Target database (Geosoft Oasis Montaj™ format) of contractor picks for each sensor type, including reacquisition.

4.4 Reacquisition

An ERT reacquisition team will perform reacquisition of the interpreted geophysical anomalies using the dig sheet. The Robotic Total Station (RTS) positioning system will be used by reacquisition personnel to validate this system for the reacquisition phase of the project. A hand held sensor (Vallon VMH series, Minelab Explorer) or equivalent will be used by personnel to pinpoint the target specified on the dig sheet, and these coordinates will be stored by the respective positioning system and/or recorded on the dig sheet. The coordinates of the reacquired position will be compared to the interpreted coordinates on the dig sheet to ensure the requirements in DID MR-005-005 are achieved; the requirement states that 95% of the reacquired locations shall fall within a 1 meter radius of the target location as specified by the interpreter on the dig sheet and 95% of all excavated items must lie within a 35cm radius of their mapped surface location as marked in the field after reacquisition.

Figure 4-1. GPO Test Plot Location



Geophysical Test Plot Layout

Figure 4-2. GPO Test Plot Seed Locations

Table 4-1. Descriptions and Locations of Seeded Items with the GPO Test Plot

Test Plot Corner Coordinates

Grid Corner	X (WGS84_UTM Zone15N-meters)	Y (WGS84_UTM Zone15N-meters)
SW	717342.541	4680391.943
SE	717354.329	4680401.219
NE	717317.227	4680448.372
NW	717305.439	4680439.097

Survey Location of Seeded Inert Items

X (WGS84_UTM Zone15N-meters)	Y (WGS84_UTM Zone15N-meters)	Target	Depth (Ft)	Azimuth (deg) CW from North	Inclination (deg) 0 deg Horz 90 deg Vert	Description
717330.288	4680414.041	1	0.10	0	0	20 mm projectile
717342.260	4680399.160	2	3.50	45	0	81 mm projectile casing
717342.739	4680403.396	3	0.50	0	0	37 mm projectile
717333.394	4680410.118	4	0.30	0	0	20 mm projectile
717342.449	4680408.135	5	1.50	90	0	75 mm projectile
717336.529	4680406.251	6	0.50	0	0	20 mm projectile
717337.739	4680413.197	7	2.00	0	90	81 mm projectile
717339.863	4680413.456	8	0.40	0	0	Mk- 2 hand grenade
717339.614	4680402.348	9	0.70	0	0	20 mm projectile
717350.870	4680403.390	10	4.00	180	30	155 mm projectile
717333.121	4680418.694	11	1.00	0	90	3.5" Rocket Fuse
717332.828	4680422.021	12	1.80	90	20	60 mm projectile
717327.374	4680421.311	13	2.00	45	45	75 mm projectile
717323.185	4680421.230	14	0.50	0	0	3.5" Rocket Fuse
717329.463	4680426.783	15	1.00	0	0	60 mm projectile
717325.212	4680428.098	16	2.50	180	0	75 mm projectile
717320.396	4680426.075	17	0.80	0	0	Mk- 2 hand grenade
717324.948	4680431.719	18	0.50	0	0	60 mm projectile
717320.103	4680431.792	19	1.20	180	70	3.5" rocket w/head
717316.824	4680432.480	20	2.00	45	20	60 mm projectile
717320.192	4680438.330	21	1.00	0	0	60 mm projectile
717312.673	4680434.977	22	1.30	45	30	37 mm projectile
717315.343	4680437.627	23	0.80	0	45	37 mm projectile
717312.543	4680439.771	24	2.50	0	20	3.5" rocket w/head
717316.273	4680442.227	25	2.50	0	45	81 mm projectile

Table 4-2. Geophysical QC Schedule

Test #	Test Description	Power On	Beginning of Day	Beginning and End of Day	1st Day of Project	Repeat Two Lines on Each Grid
1	Equipment Warm-up	X				
2	Personnel Test		X			
4	Vibration Test (Cable Shake			X		
5	Static Background and Static Spike			X		
6	6 Line Test				X	
7	Repeat Lines					X

5.0 GEOPHYSICAL INVESTIGATION APPROACH

5.1 Investigation Areas

SVAD-050 can be divided into three different areas based on historic usage and the investigation approach defined in this Work Plan. These areas are described in the main Work Plan document as the Open Detonation (OD) Area, the Open Burn (OB) Area, and Other Areas (roads). A total of approximately 7 acres of land will be geophysically investigated including approximately 5 acres of the OD area, 0.8 acres of the OB area, and 1 acre of the Other Areas including SVAD-050 roads. Figure 5-1 shows the locations of geophysical investigation areas. Only areas covered by less than 2 inches of water, and those areas that have been surface swept for MEC and metals will be geophysically surveyed. The geophysical coverage of SVAD-050 may be affected by regular flooding that occurs in the OD Area.

5.2 Grid Method Surveying

A 100 x 100-foot grid system will be established over all of SVAD-050 both for reference and navigation during geophysical surveys, the grid can be seen on Figure 5-1. The grid will be installed by an Illinois licensed surveyor. All surveying activities necessary to perform geophysical mapping will be performed in accordance with DID MR-005-07.01, Location Surveys and Mapping Plan. Each grid planned for geophysical investigation will then be cleared of surface vegetation and surface metals following procedures outlined in Appendix C of the Work Plan (MEC Investigation Plan). Geophysical investigations will follow on a grid-by-grid basis.

5.3 Geophysical Instrumentation

Geophysical instrumentation will include the Geonics EM61-MK2 metal detector (EM61-MK2), and Leica TPS 1200 RTS. The EM61-MK2 is a Time Domain Electromagnetic (TDEM) system, which generates 150 electromagnetic (EM) pulses per second from an air-cored copper transceiver coil. After each pulse, secondary EM fields are induced briefly in moderately conductive soils and for a longer time in metallic objects. Between each pulse, the EM61-MK2 waits until the response from the conductive earth dissipates and then measures the prolonged buried metal response. This response is recorded in millivolts (mV). By sensing only the buried metal response, the EM61-MK2 detects metallic targets that might otherwise be missed. The EM61-MK2 measures multiple time gates (216, 366, 660, and 1266 μsec) to provide a more complete measurement of the response decay rate. The EM61-MK2 can record up to 12 records per second and four time gates per record. The transceiver coil is 1.0 by 0.5 meter and sits on wheels 40.5 centimeters above the ground.

The EM61-MK2 is capable of either measuring a differential voltage, calculated as the voltage difference between the top and bottom coils at a single time gate, or measuring voltages at four different time gates using only the bottom coil. The differential voltage is useful in reducing noise in data, while the readings at four time gates are useful in distinguishing the responses from different ordnance items. Stacking data from all four channels can also reduce the interference of noise. Therefore, for this project, data will be collected at four channels using the bottom coil.

It is anticipated that RTS navigation will be employed for the geophysical surveys in SVAD-050.

The RTS system includes a total station instrument that is set up on a first order survey benchmark, a mobile prism target, and a remote data logger. As part of this investigation, a 100x 100-foot grid of control points will be established an Illinois Licensed Surveyor for use as RTS base stations. The total station instrument combines a theodolite (a device for measuring very precise angles) and a laser-based electronic distance-measuring device (EDM). The EDM measures the distance from the instrument to the target by sending out a laser beam, which is reflected back to the total station by a prism. Using timing measurements, the RTS calculates the distance traveled by the beam.

The RTS measures angles and distances from the total station to the point surveyed. These angles and distances are then used to calculate the actual positions (x, y, and z, or northing, easting, and elevation) of the surveyed point. The readout is continuous so that the angles can be checked at any time.

Automatic tracking of the prism by the total station is based on a line of sight. Because the system tracks the prism at the survey speed, it is important to maintain a steady forward speed. If the line of site is lost briefly, the system assumes a constant speed of the target and if the target maintains the same speed the efficiency of the system to re-establish prism lock will be high. Once line of sight is interrupted and the laser-to-prism lock is lost, the operator stops and pauses the geophysical data recording until the prism lock is re-established. For short interruptions, e.g. 1 second, positional data are interpolated between the last locked point and the first point once the lock is re-established. The RTS system is ideal for use in areas where tree canopy blocks GPS signal.

Data will be collected on a grid-by-grid basis using a 3-foot line spacing maintained using both real-time position display Trackmaker® software, as well as 300-foot fiberglass measuring tapes and marking paint. A pace of less than 3 miles per hour (mph) will be used to maximize data density. Files will be saved using the date followed by grid reference and incremental letters. For example, the first file collected in Grid B3 on June 10 2008 would be 061008B3A, followed by 061008B3B and 061008B3C etc.

Notes will be recorded in the field log book including information such as dates/times, grids surveyed, equipment operator, battery voltage, QC files, field sketches and field conditions.

5.4 Data Processing, Analysis and Interpretation

5.4.1 EM61-MK2 Data Processing and Target Selection

Geonics DAT61MK2® software is used to download the EM61-MK2 data to a laptop in the field. The data is converted from the .R61 binary format into ASCII format .M61 files. The ASCII files are then reviewed using Notepad® and DAT61MK2®. The data are reviewed for data gaps. After this preliminary data review is complete and the data set passes all review criteria, the file is exported to *Geosoft* .xyz format. Should any data gaps or system malfunctions be detected during the preliminary data review, specific areas will be flagged for immediate re-collection.

Reviewed EM61-MK2 data is then imported to Geosoft Oasis Montaj® and processed using the following procedures:

- The data is converted to NAD83, Illinois State Plane coordinates in US survey ft;
- Latency corrections are performed based on instrument latency determined from the Six Line Test (conducted during GPO investigations) using the UCELATENCY application. Verification of proper latency corrections is made by reviewing maps for "chevron effects";
- All channels are leveled to a common 0-mV median baseline using the UX-detect drift correction algorithm;
- Data are reviewed for completeness as defined in the DQOs (Section 6.2). Line paths are plotted to evaluate across-track sampling and verify that no data gaps are present. A velocity map is generated to locate any areas where collection speed is above 3 mph;
- Individual channels as well as stacked channels 1-4 are gridded using the minimum curvature algorithm with a cell size of 0.3 ft, the project geophysicist may adjust gridding parameters accordingly based on sample density;
- A series of color maps are produced from the gridded data;
- Line paths are posted over the mapped data, and reviewed again for coverage completeness.

The following procedures will be followed for EM target selection:

- A target threshold value is selected that would minimize the amount of target picks without excluding items of interest. This target threshold value will be based on the results of the GPO investigation, and be reported in the GPO letter report. The threshold is established based on an evaluation of the noise levels and the detection of the known location seed items in order to maximize detection performance while minimizing the numbers of false positives being detected. Targets are automatically selected using the Blakely method within UX-Detect®.
- A decay curve criteria of channel 1>channel 2>channel 3 >channel 4 responses are used to differentiate targets of interest from other signature sources. The target locations are reviewed and manually adjusted during target QC. If the targets are caused by cultural influences, they are deleted. Additionally, the data are analyzed using the UX-Process® "Calculate signal strength, SNR, and size" routine. Targets will then be manually reviewed and adjusted as needed. Co-located anomalies may be merged if they fall within a 2-foot search radius. Large or continuous anomalies displaying the geophysical characteristics of burial pits or trenches will be identified on maps as polygons.

All corrected geophysical data associated with anomaly locations will be exported to a target database. Throughout the geophysical survey, field personnel will use logbooks to record observations such as variances in the background interference/noise when collecting data, and/or notable changes in soil characteristics. Such observations will provide valuable insight during the selection of anomalies in areas where there are significant variations in background interference/noise.

5.4.2 Target Lists

A final target list will be generated for each investigation grid, in which the following information will be included:

- Unique target ID;

- Easting and Northing positions;
- Response amplitude for individual EM channels, stacked channels and EM size; and
- Recommendations by the Project Geophysicist.

As test pits will be excavated in areas of high anomaly concentration for this investigation, rather than numerous single-item targets, the Project Geophysicist will make recommendations for a total of six test pit locations. Each test pit will be excavated to 4 ft below ground surface (bgs) and will cover an area sufficient to recover any concentration of munitions identified by the Project Geophysicist, with a minimum surface area of 25 ft^2 and a maximum of 50 ft^2 per test pit. At least one test pit will be excavated in each of the OD and OB areas. Additional single-target geophysical anomalies may be excavated if the six test pits are deemed insufficient by the Project Geophysicist to establish an understanding of the nature of geophysical anomalies.

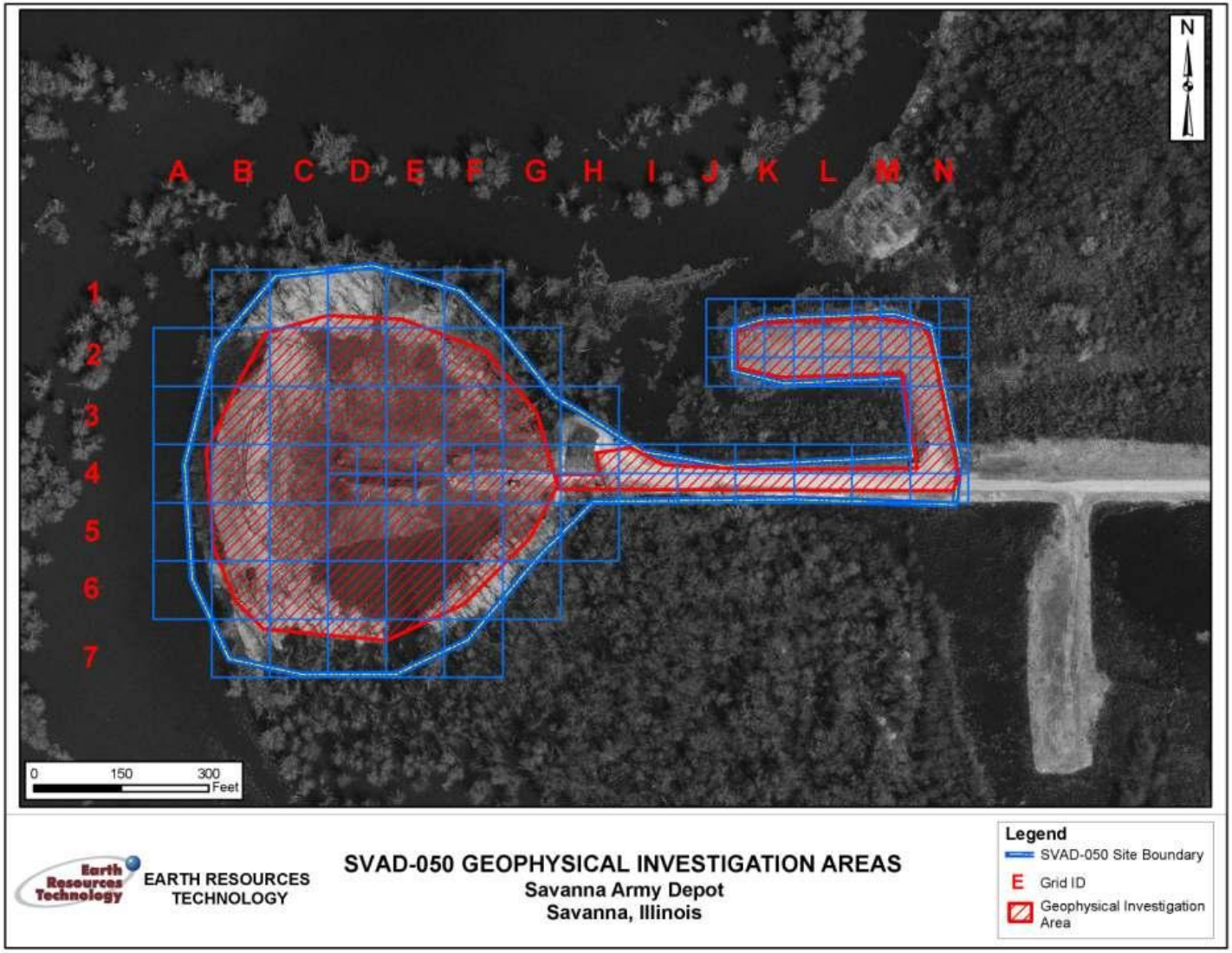


Figure 5-1. Locations of Geophysical Investigation Areas

6.0 PRODUCTION QUALITY CONTROL

6.1 Instrument Function Checks

ERT will perform standard instrument function tests as described in Section 4.3. Each of these tests, with the exception of the Six Line Test will be performed twice daily, once before and once after data collection. The Six Line Test will be performed once at the beginning of the project, during GPO data collection.

6.2 Production Data Quality Objectives

The following DQOs will be followed for data collection and processing within SVAD-050.

- Processing: All leveling and/or filtering routines that are applied to datasets will be evaluated, on a dataset-by-dataset basis, to confirm that those routines do not alter the nature of the original measured response.
- Navigation: Navigation may be evaluated by seeded Quality Assurance (QA) items and positioning relative to pre-established control monuments. Seed items may be randomly placed by the USACE.
- Mean Speed: Data acquisition rates should be consistent with the achieved rates during the GPO. Maintain mean speed less than or equal to 3 mph.
- Along Track Sampling: Less than or equal to 0.6 ft.
- Across Track Sampling: Less than or equal to 3.0 ft.
- Data Leveling: Use consistent parameters and processing methods for all datasets on grid by grid basis.
- Signal above Background: Processing not alter signal above background by more than 5% or 5 mV, whichever is less (i.e., the difference between background and peak response).
- Anomaly Selection: Do not rely on the automated system only for anomaly selections. Verify that anomaly selections are reasonable for identifying anomalies consistent with individual MEC items or burial pits. A decay curve criteria of channel 1>channel 2>channel 3 >channel 4 responses are used to differentiate targets of interest from other signature sources.
- Repeatability: Subsequent to completing data collection, EM data will be recollected on at least 3% of the survey area to demonstrate navigational and response consistency.
- Blind/Seeded QA items: All blind seeded items must be detected within 2 ft.

The Geophysical Report will identify the project DQOs and data to support the attainment of the DQOs.

6.3 Corrective Actions

ERT will adhere strictly to the procedures described in this GIP to perform the geophysical investigations. Based on the QC checks described in the previous section, if it appears that a particular geophysical method, instrument, or procedure is not generating meaningful results or

achieving the project goals, ERT will convene a review team consisting of ERT's Project Geophysicist, Project Manager, and the USACE to investigate the cause and recommend corrective actions. If minor process modifications can be made or equipment substituted to achieve the goal, ERT will take action to make these amendments, and the Army will receive written notification of all actions taken. If the instrument or process cannot be corrected to achieve the desired DQOs, ERT will cease using that method/instrument and will make recommendations to the USACE. These recommendations may include modifications to the Work Plan. ERT will implement the amended plan upon approval from the USACE.

7.0 DATA MANAGEMENT

All electronic deliverables including Draft and Draft Final reports, as well as raw and processed geophysical data, will be submitted to the USACE on compact disc (CD). All data and documents will be maintained in separate investigation grid subfolders and will remain backed-up on the ERT internal network.

SVAD-050 project data will have the following general organization:

- Plans – Work Plans
- Reports/Deliverables
- Geophysical Prove-out (GPO)
- Field Data – Daily field data, QC, preprocessing and review
- Target Data – Target re-acquisition, intrusive sampling results, and dig clearance data

A complete, bound Geophysical Report will be submitted along with electronic deliverables as part of the SVAD-050 Remedial Investigation Report.

8.0 REFERENCES

US Army Corps of Engineers (USACE), 2002. *Location Surveys and Mapping Plan.* DID MR-005-07.01.

USACE, 2004. *Personnel/Work Standards* DID MR-025.01

USACE, 2007. *Geophysics* DID MR-005-05.01

APPENDIX C
MEC INVESTIGATION PLAN

FINAL
MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) INVESTIGATION PLAN FOR REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION AREA (SVAD-050), SAVANNA ARMY DEPOT SAVANNA, ILLINOIS

Prepared for:

U.S. Army Corps of Engineers
Louisville District

Contract W912QR-08-D-0012
Delivery Order 0001

Prepared by:

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September 2009

FINAL
MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) INVESTIGATION PLAN
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LIST OF ACRONYMS AND ABBREVIATIONS

ATF	U.S. Bureau of Alcohol, Tobacco, Firearms, and Explosives
BEC	BRAC Environmental Coordinator
bgs	Below Ground Surface
BIP	Blown in Place
BRAC	Base Realignment and Closure
CFR	Code of Federal Regulations
CIH	Certified Industrial Hygienist
DA	Department of the Army
DID	Data Item Description
DoD	Department of Defense
DOT	Department of Transportation
EED	Electro-explosive Device
EMR	Electromagnetic Radiation
EOD	Explosive Ordnance Disposal
ERT	Earth Resources Technology, Inc.
EZ	Exclusion Zone
GIP	Geophysical Investigation Plan
GPO	Geophysical Prove-out
GPS	Global Positioning System
HASP	Health and Safety Plan
MEC	Munitions and Explosives of Concern
MFR	Maximum Fragmentation Range
MGFD	Munition with the Greatest Fragmentation Distance
MSD	Minimum Separation Distance
OB	Open Burn
OB/OD	Open Burn/Open Detonation
OD	Open Detonation
OSHA	Occupational Safety and Health Administration
PETN	Pentaerythritoltetranitrate
QC	Quality Control
RDX	Cyclotrimethylenetrinitramine
RF	Radio-Frequency
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
SHA	Safe Holding Area
SMS	Safety Management System
SOW	Statement of Work
SUXOS	Senior UXO Supervisor
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
TNT	Trinitrotoluene
TSD	Team Separation Distance
UHF	Ultra High Frequency
USACE	United States Army Corps of Engineers
USAESCH	United States Army Engineering Support Center, Huntsville

UXO	Unexploded Ordnance
UXOQCS	Unexploded Ordnance Quality Control Specialist
UXOSO	Unexploded Ordnance Safety Officer
VHF	Very High Frequency

1.0 INTRODUCTION

1.1 Purpose

The objective of the Task Order is to evaluate the nature and extent of potential contamination at the Savanna Army Depot Activity (SVDA), Site 50 (SVAD-050) from past practices, determine if the potential contamination poses threat to human and ecological receptors, and develop a feasibility study to evaluate and select an alternative to minimize any potential threats. As a former Munitions and Explosives of Concern (MEC) Open Burn (OB) and Open Detonation (OD) disposal area, SVAD-050 contains MEC waste that is both a focus of investigations and a safety concern during field efforts. This document summarizes the methods that will be employed to support geophysical and environmental MEC sampling including escort and avoidance, MEC removal, intrusive investigations and MEC disposal.

1.2 Munitions History

SVAD-050 has been used since approximately 1936 for the disposal of various explosives, such as trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), and pentaerythritoltetranitrate (PETN) by venting, as well as demolition of conventional MEC ranging in size from 20 mm to 155 mm artillery rounds. SVAD-050 covers approximately 7 acres. Three distinct areas within SVAD-050 have been identified based on historic use and the investigation approach outlined in the Work Plan. These areas are the OD Area, the OB Area, and other Areas (i.e., the roads between the two areas). Each of these areas is identified in the main Work Plan document.

2.0 MEC INVESTIGATION ACTIVITIES

The following activities will be performed during SVAD-050 Remedial Investigation (RI) by qualified unexploded ordnance (UXO) technicians.

2.1 Vegetation Removal

Earth Resources Technology, Inc. (ERT) will perform all brush clearing activities using UXO team personnel. Vegetation clearing will be required in all areas where geophysical investigations are planned. This task will take place after the land survey crew has established grid control points over the entire site. Grid control points will be used as guidance when removing vegetation from areas marked for MEC surface clearance and geophysical investigation. Areas planned for geophysical investigations (and vegetation clearance) are identified in Figure 5-1 of the Geophysical Investigation Plan (GIP) (ERT, 2009a), most areas marked for clearing are covered with grasses, however some brush and small saplings are found in parts of the OD Area. Only area that is covered by less than 2 inches of water will be cleared of vegetation. Regular flooding of the OD area may affect the amount of land that can be cleared of vegetation. The vegetation clearing crew will consist of a UXO Technician II and a UXO Technician I. One crew is likely to be required, based on current site conditions, for brush clearing activities. A brush-hog, or similar device, will be used to mechanically remove vegetation. The operator of the brush hog will follow a path that has been visually inspected by a UXO Technician utilizing hand-held brush clearing equipment such as chain saws or machetes. All brush clearing safety guidelines established in the Site Safety and Health Plan (SSHP) (ERT, 2009b) and in United States Army Corps of Engineers (USACE) Engineering Manual (EM) 385-1-1 (2003) will be followed. Cut vegetation will be mulched and placed, per direction from the SVDA Base Closure and Realignment (BRAC) Environmental Coordinator (BEC).

2.2 Escort

During all field activities at SVAD-050, UXO avoidance support in the form of a UXO Technician II escort will be required of any non-qualified UXO personnel working in areas that have not been surface cleared of MEC. This includes groundwater sampling, soil sampling, and land surveying/grid establishment. Work may be performed without escort if the area has been previously cleared of MEC and if intrusive activities are not planned.

2.3 Surface MEC Removal

After a control grid and vegetation clearing have been performed at the site, all areas planned for geophysical investigation will be surface cleared of all MEC, MD and metallic debris. Areas planned for geophysical investigation include the OD area, the OB area, and the roads between the two areas. Only areas covered with less than 2 inches of water will be surface swept for MEC and metals. Because regular flooding of the OD Area may impact the amount of land that can be surface swept, conducting the sweep during flooding will be avoided. No MEC removal or intrusive activities will be performed prior to mobilization of the Senior UXO Supervisor (SUXOS) and the establishment of a command post with reliable communications.

All surface metals and MEC material will be removed by a single team consisting of one UXO Technician III, one UXO Technician II, and five UXO Technician I personnel. The methods for removal will be as follows:

- Lanes will be established using the grid control points previously staked by an Illinois licensed surveyor and using fiberglass measuring tapes/ropes as guides. Seven-member teams will work grid by grid to identify and remove all surface metals and MEC related materials. Lanes will be walked while performing both a visual inspection and a sweep using Schonstedt 52CX magnetometers.
- Each seven-member team will be comprised of one UXO Technician III, one UXO Technician II, and five UXO Technician I personnel. The Technician II and I personnel will work in teams of two to identify and remove MEC-related materials. The Technician III will oversee the team's activities, monitoring safety, quality control (QC), and assisting with identification.
- Identification will follow the methods detailed in Section 4.3. Materials safe for handling and transport will be temporarily stored in approved secure storage containers as detailed in Section 4.3.
- A log will be maintained by each team's UXO Technician III. After each grid has been cleared it will be added to a list of grids ready for geophysical investigation.

2.4 Test Pit Excavations

Six test pits will be excavated in areas of high concentrations of geophysical anomalies. At least one test pit will be excavated in the OD area and one test pit will be excavated in the OB area. Additional single-target geophysical anomalies may be excavated if the six test pits are deemed insufficient to establish an understanding of the nature of geophysical anomalies.

Test pit locations will be located using Global Positioning System (GPS) coordinates received from the site geophysicist. Each test pit will be excavated to four feet (ft) below ground surface (bgs) and will cover an area sufficient to recover any concentration of munitions identified by the site geophysicist, with a minimum surface area of 25 ft^2 and a maximum surface area of 50 ft^2 per test pit. Test pits will be hand excavated by teams of three made up of one UXO Technician III and two UXO Technician I personnel. Additional individual-target anomalies may be excavated if deemed necessary.

During test pit excavation, soil screening will be performed. Using a handmade screening unit (a base 4 ft wide by 4 ft long with legs approximately 4 ft high and covered by either secured ¼ inch metallic or ¼ inch wire screening materials), soil will be placed on the screening unit initially using shovels. Physical screening of soils will be conducted by hand. MEC materials remaining on the screen will be inspected for explosives or explosive constituents and placed in appropriate containers. The inspected soil will be staged at the screening site and will be returned to the excavation once intrusive operations are completed.

For each MEC item recovered and identified, the following information will be documented:

- Depth
- Position
- Attitude
- Condition (fired/unfired)
- Offset from original coordinates
- Identification

- If nothing is found, the anomaly must be reported as a "No Find"

Should the excavation team locate an area suspected to be a burial pit, excavation will cease and the SUXOS and the USACE Safety Specialist will be notified.

3.0 QUALITY CONTROL/QUALITY ASSURANCE

An existing geophysical prove-out (GPO) will be utilized for daily equipment function testing. Daily standardization check of the geophysical sensors will be conducted in accordance with Data Item Description (DID) MR-005-05.01 (USACE, 2007). These activities will be documented in the team leader's logbook and/or field data tracking forms.

4.0 MEC MANAGEMENT

4.1 Site Control

Access to SVAD-050 and range fan area at the Savanna Army Depot is controlled by gated access from a perimeter access road. Access to the site area from the Mississippi River is prevented by a buoy system around the perimeter of the area. In addition, access via the river would be difficult due to the shallow slough system around the site. An exclusion zone (EZ) including a 2,577-foot radius will be maintained around all MEC activities described in Section 7.0, based on the munition with the greatest fragmentation distance (MGFD).. The perimeter of the EZ will be marked on all intersecting roads using barricades. The nearest road (River Road) outside of SVAD-050 is approximately 2,000 ft from the eastern perimeter of SVAD-050. Access to this road is restricted via locked gate,; however barricades and/or guards will be placed along the road during all MEC activities. Figure 4-1 shows an EZ buffer surrounding SVAD-050.

A command post will be established at Building 1100 prior to beginning any intrusive activities.

4.2 Personnel Responsibilities

All UXO technical staff will be trained and meet the standards set forth by DID OE-025.01 (USACE, 2002a). The organizational chart provided in Figure 4-2 describes the relationships between UXO personnel on this project.

4.2.1 SUXOS

The SUXOS, Mr. Michael Garner, will be responsible for all field activities related to MEC operations and ensure that all field operations are in compliance with the SSHP (ERT, 2009b). The SUXOS will report to the Project Manager and coordinate with the UXO Safety Officer (UXOSO)/UXO Quality Control Specialist (UXOQCS). The SUXOS will be assigned the following authority/responsibilities:

- Serves as technical lead for MEC operations;
- Implements and enforces the Safety and Health Plan (SSHP), reporting violations to the Project Manager and UXOSO;
- Establishes work zones and controls access to those zones;
- Assists in the continued development of the SSHP and other site and health procedures;
- Assists the UXOSO in conducting injury/incident/near miss reporting and investigation for MEC-related occurrences;
- Enforces use of the "buddy" system;
- Restricts personnel exhibiting symptoms of alcohol/drug use or illness from site activities and continually monitors site personnel for signs of chemical exposure or physical stress;
- Maintains site safety and monitoring logs; and
- Notifies and coordinates off-site emergency and medical response agencies.

4.2.2 UXOSO/UXOQCS

For this project, the duties of the UXO Safety Officer and Quality Control Specialist will be performed by one person, Mr. George Payne. The UXOSO/UXOQCS will report directly to the

Project Manager and coordinate activity with the SUXOS. The UXOSO will be the primary person responsible for day-to-day health and safety during the project. The UXOSO will be assigned the following responsibilities:

- Provides implementation/oversight of ERT safety and health matters.
- Ensures compliance with ERT's Corporate Safety Plan and the SSHP (see RI/FS Work Plan, Appendix A).
- Confirms suitability for work of contractor and subcontractor personnel, based on Occupational Safety and Health Administration (OSHA) and site-specific medical and training requirements.
- Conducts visitor orientation.
- Conducts on-site safety orientation and operational review on the first working day.
- Maintains a list of training records and expiration dates of applicable training at the work site for all on-site personnel (including any government workers).
- Ensures copies of all documents listed in the SSHP are readily accessible by site personnel and visitors (these will be on-site in hard copy form when practicable and feasible).
- Conducts daily safety meetings.
- Conducts and documents daily safety inspections, weekly safety audits, and self assessments as required by ERT SSHP and this plan.
- Maintains the safety, training, equipment maintenance, and visitor logs.
- Consults with the project Certified Industrial Hygienist (CIH), as needed, to ensure all potential hazards are addressed appropriately.
- Enforces the ERT Alcohol/Drug Abuse Policy.
- Trains personnel on the emergency action plan requirements for the site.
- Ensures prominent display of descriptions and maps associated with local hospital and emergency evacuation routes.
- Enforces the use of the "buddy" system.
- Ensures first aid kits are on-site and adequately stocked
- Provides oversight and serves as the interface for subcontractor safety and health representatives.
- Conducts injury/illness/incident/near miss reporting and investigation.
- Maintains a list of medical-specific information for all site personnel (e.g., blood type, allergies, medications, high blood pressure).
- Ensures compliance with the written Work Plan;
- Conducts required inspections and surveillances;
- Ensures reporting procedures are being followed;
- Identifies and addresses conditions both negatively and positively affecting the project, and reports those conditions; and
- Stops work that is not in compliance with the contract.

4.2.3 UXO Technician III

UXO Technician IIIs will report to the SUXOS. They will be responsible for providing direct supervision to and ensuring the safety of personnel assigned, both UXO-qualified and non-UXO qualified. They will ensure that all work conforms to, and that all personnel comply with, the Work Plan, including the HASP, and applicable federal, state, and local regulations. The Technician IIIs may conduct additional safety meetings and training as needed. They will report all safety violations and injuries to the SUXOS and the UXOSO. Technician IIIs will be assigned as team leaders supervising UXO Technician IIs and UXO Technician Is and/or non-UXO personnel.

Only a UXO Technician III will fill the team's UXO Team Leader position for all MEC dig teams. The UXO Technician III will be a graduate of either the Naval Explosive Ordnance Disposal (EOD) School or Army Bomb Disposal School at Aberdeen, Maryland, or have completed a Department of Defense (DoD) certified UXO training program and have at least 10 years of experience. The UXO Technician III will be able to fully perform all the functions of UXO Technician II, as well as documentation of MEC finds and direction of field crew. Additional responsibilities include:

- Post individuals at entry points (if required);
- Serve as Team Lead for MEC surface clearance and test pit excavation teams, overseeing up to six individuals on such teams.
- Construct appropriate Engineering controls in accordance with "Use of Sandbags for Mitigation of Fragmentation and Blast Effects Due to Intentional Detonation of Munitions," CEHNC-ED-CS-S-98-7 (USACE, 1998), if required;
- Assign team members to specific demolition duties;
- Ensure the area is clear prior to capping in; and
- Check the area following each shot or series of shots.

4.2.4 UXO Technician II

UXO Technician IIs will comply with the provisions of the SSHP, Work Plan, and applicable federal, state, and local regulations. They will report to their assigned UXO Technician III for performing duties as a member of functional teams.

UXO Technician IIs will be experienced in UXO remediation/range clearance operations, and have project-specific ordnance training. The UXO Technician II will be a graduate of either the Naval EOD School or Army Bomb Disposal School at Aberdeen, Maryland or have completed a DoD-certified UXO training program and have at least 5 years of experience. The UXO Technician II will be able to perform all the functions of a UXO Technician I as well as provide UXO escort for non-UXO qualified personnel.

4.2.5 UXO Technician I

UXO Technician Is will be graduates of a DoD-certified training program and will be able to perform the following functions:

- Properly use and maintain UXO location equipment, to include, but not be limited to, locators and GPS units;
- Positively identify surface items as MEC;

- Conduct subsurface anomaly reacquisition utilizing Schonstedt or Vallon locators; and
- Intrusively investigate subsurface anomalies.

4.3 MEC Storage, Transportation and Disposal

4.3.1 MEC Identification

MEC will be identified and placed in four categories. Cultural debris will also be collected and maintained separately as either refuse or scrap metal. Items that require demilitarization will be done in accordance with DoD 4160.21-M, Defense Materiel Disposition Manual (DoD, 1997). The following four categories of MEC will be identified and treated uniquely:

- Ordnance Related Scrap
- Raw Explosive
- Unfired Live – can move
- Fired Live – blown in place (BIP)

As MEC is located it will be documented on the working map and entered into the MEC Accountability Log (Attachment 1).

A detailed accounting of all live/suspected MEC items encountered during the MEC removal action will be accomplished. The ERT UXO Accountability Log will be completed on each live/suspected UXO or MEC item encountered. This accounting will include:

- A unique Identification Number
- Transect Location
- Nomenclature (by M number)
- Whether item appears to have been fired
- Fuze Description (include M number)
- Fuze Condition
- Additional comments, if required.
- An item will be considered to have been fired if there are rifling marks on its rotating bands.

Each type of live or suspect MEC item encountered will be identified using a unique numerical identifier, such as PR – G1 – 0001 for first live/suspect type item (0001) encountered in grid G1 at the project site.

Photographs of live or suspect MEC items will be taken for documentation purposes. A ruler or some similar item, to show scale, will be placed adjacent to the item. The photographer needs to remember these photographs will be utilized in the final report; thus, a focused, well thought out photograph is necessary. The identification process for each type of MEC item is detailed in the following sub-sections.

If an unidentifiable MEC is found, a request for EOD support will be made through the SVDA BEC and the Louisville District Project Technical Manager will be notified. ERT will secure the item until relieved of the responsibility by the appropriate Government representative.

4.3.1.1 Scrap

Items will first be identified by a UXO Technician I or II, as they are located. If the item is determined to be scrap metal, it will be collected and positively identified as scrap by the UXO Technician III Team Leader by the end of the same day. Scrap will then be placed in an appropriate bulk container until removal from the site.

Before scrap material leaves the site, the SUXOS and UXOSO/UXOQCS will perform an additional inspection of the scrap. Once the inspection has taken place, the SUXOS will prepare a DD Form 1348-1 (Attachment 2) signifying that the material has been inspected. The materials will be loaded for transportation to a holding area, the location of which will be coordinated and approved by the USACE and the SVDA BEC.

4.3.1.2 Raw Explosive

Items identified as raw explosive by the UXO Technician I or II will be immediately inspected by the UXO Technician III Team Leader. Raw explosive positively identified at this point can be safely transported in a secure sealable container for co-location and subsequent disposal. Both the SUXOS and UXOSO/UXOQCS will inspect all material identified as raw explosive prior to processing as described in Section 4.3.4.

4.3.1.3 Unfired Live

Identified unfired live ordnance must be immediately inspected by the UXO Technician III Team Leader. The SUXOS and UXOSO will then jointly determine if an MEC item is live or suspected live before any removal action is attempted. The SUXOS will make the final decision on whether unfired live MEC is safe to transport for co-location and subsequent demilitarization, or should be blown in place (BIP). All available data sources will be consulted prior to this determination. Unfired live MEC will be demilitarized following disposal methods described in Section 4.3.4.

4.3.1.4 Fired Live

Any item identified as fired live ordnance must be immediately inspected by the UXO Technician III Team Leader. Items identified as fired live ordnance will then be inspected in place by the SUXOS and UXOSO. Armed and fired live items deemed as too hazardous to move will be marked for BIP disposal, and will be safely covered, marked and have the position recorded using GPS. ERT will secure the item until such time as BIP procedures can be completed.

4.3.2 MEC Storage

All MEC material will remain onsite until it has been demilitarized and certified by the SUXOS, and UXOSO (see Section 4.3.4). MEC materials deemed unsafe to move will be BIP. MEC material deemed as safe to move will be transported for later consolidated disposal in the OD area. MEC that has been deemed safe to move will be stored in either a temporary explosive storage vault to be located just off the eastern portion of the OD area, or in the Personnel Bunker on the eastern edge of the OD area, based on the preference and approval of the SVDA BEC. In either case, the storage facility will be locked and secured. Since access to the site is limited to authorized personnel, a security guard will not be necessary if materials are kept in locked storage.

ERT will not establish a Safe Holding Area, as all live or suspected-live MEC will be BIP or if deemed acceptable to move, transported for secure storage in the storage vault/Personnel Bunker.

4.3.3 MEC Transportation

ERT does not anticipate transporting any MEC items off site. MEC that is deemed safe for transportation may be transported within SVAD-050 to the OD area for demilitarization. All MEC transport will be in approved secure, sealable containers.

4.3.4 MEC Demilitarization

All MEC demilitarization/disposal will occur on the last day of the work week, typically Friday. This includes both consolidated disposal shots and demilitarization procedures. All demilitarization activities will take place within the OD project area, between the existing berms, with the exception of items marked for BIP operations. All demolition activities will be conducted in accordance with the SSHP and MEC demolition requirements. Demolition activities will be in compliance with:

- EP 385-1-95a, USACE Basic Safety Concepts and Considerations for Munitions and Explosives of Concern (MEC) Response Action Operations (USACE, 2004).
- DoD 6055.9-STD, DoD Ammunition and Explosive Safety Standards (DoD, 2008).
- Technical Manual (TM) 60A-1-1-31, Explosive Ordnance Disposal Procedures (Dept. of the Army, 1969).

If MEC is encountered that cannot be moved due to its condition and the location prevents disposal in place, the SVDA BEC will be notified. If a Government representative is not on site, the Louisville District Project Technical Manager will be notified for direction. ERT will maintain security of the item until relieved of that responsibility by appropriate Government representative, or until a decision is made to allow ERT to proceed with BIP procedures using engineering controls.

Both BIP and co-located items to be demilitarized will be explosively detonated using electric firing methods. The electric firing method allows for full control of the timing of explosive detonations, providing last-minute control in the case of safety breaches.

4.3.4.1 Electric Firing Procedures

Electric firing will follow all guidelines and safety protocols outlined in TM 60A-1-1-31. General firing procedures will be as follows:

- Establish a safety exclusion zone (see Explosive Siting Plan-Section 6) for demolition activities. Access to the exclusion zone will be restricted using barriers and guards, if necessary.
- Place supplemental charge on each MEC item to be demilitarized.
- Connect detonation cord to supplemental charge
- Encapsulate MEC items in sand bag enclosure to minimize fragmentation
- Un-shunt cap leads and connect to appropriate testing device
- Test cap(s) for continuity and re-shunt leads.
- Tape caps to final detonation cord lead (do not bury electrical caps).

- A sand bag may be placed gently over the caps and detonation cord to prevent movement
- Perform a security check around the demolition area to ensure no unauthorized individuals have entered the exclusion zone.
- Once the UXOSO is satisfied that the area is secure he will order the demolition supervisor (UXO Technician III) to connect the firing circuit to the main charge and perform a circuit check.
- Once the circuit check is complete, a final check of the demolition area and the exclusion zone for unauthorized personnel is conducted.
- If the area is still secure, the UXOSO will authorize the demolition supervisor to conduct the MEC disposal operation.
- The demolition supervisor will yell in each of the four points of the compass "Fire in the hole" and repeat this four times on the radio. After the last "Fire in the hole" the demolition supervisor or a team member will function the firing device.
- If the demolition shot does not fire, wait 30 minutes and the demolition supervisor will investigate to determine why a malfunction occurred.

The UXO team will search the area after each firing for any remaining explosive components and loose explosives. Scattered explosive material will be carefully gathered and destroyed by detonation with the next shot. If materials are left in place, these items can create an additional explosive hazard. This search includes verifying that a secondary item is not present in the area after conducting BIP operations. The "blow-hole" will always be checked for secondary items and removal of all ordnance-related scrap and fragmentation.

The remaining ordnance-related scrap from these operations will be recovered and transported to the scrap holding area and placed into appropriate containers**.** Typically, environmental samples are collected prior to and after BIP operations to ensure that environmental impacts have not resulted. Due to the historic nature of the area as a demilitarization/demolition site, no environmental soil samples will be collected prior to or after demolition activities. All environmental sampling will be conducted as part of the RI and is outlined in Appendices D and E of the Work Plan.

Safety procedures for MEC demilitarization are included in the SSHP (Appendix A of the primary Work Plan document).

4.3.4.2 Electric Firing Hazards

Electromagnetic Radiation (EMR), static electricity, and lightning are potential hazards when using electric firing demolition. Prior to the application of detonation procedures, an EMR survey will be conducted to determine if there are any transmitting antennas of radio, radar, or other electro-magnetic-generating devices located in the vicinity.

Radio-frequency (RF) EMR consists of waves of electrical energy. These waves are radiated in a line-of-sight from the antennas of electronic devices that transmit radio, radar, television, or other communication, to include cellular telephones, or other communication or navigation radio frequency signals. Table 4-1 provides the minimum safe distance from electro-explosive devices (EEDs) and the transmitting antenna of all RF emitters. Table 4-2, provides the minimum safe

distances, which will be maintained between Mobile RF transmitters and electric blasting operations. The factors to be considered when evaluating the degree of hazard that the EMR (RF) energy represents are:

- The strength of the field (its power);
- The frequencies transmitted;
- The distance from the transmitter antenna to the ordnance; and
- The amount or type of protection available.

Lightning is a hazard to both electric and non-electric blasting caps. A strike or a nearby miss is almost certain to initiate either type of cap or other sensitive explosive elements such as caps in delay detonators. Lightning strikes, even at remote locations, may cause extremely high local earth currents, which may initiate electrical firing circuits. Effects of remote lightning strikes are multiplied by proximity to conducting elements, such as those found in buildings, fences, railroads, bridges, streams, and underground cables or conduit. The only safe procedure is to suspend all blasting activities during electrical storms and when one is impending. All blasting activities will be suspended when lightning-thunder storms are within 10 miles of the project site.

4.3.5 Disposal of Demilitarized Inert Ordnance and Scrap

All final processed material will be placed in lockable/sealable containers for security, and will be properly labeled before transport to a scrap metal processor. Alter Trading Company has been contracted by ERT to transport and process all scrap metal through a smelter or furnace. Contact information for the Alter Trading Company is as follows:

Alter Trading Company
Dubuque Processing Yard
Dubuque, Iowa 52002
Ph. (563) 583-1731

Prior to sending the scrap offsite, form DD 1348 (Attachment 2) will be signed by the SUXOS and the USACE Safety Specialist, in accordance with DoD 4160.21-M-1 (DoD, 1995).

Also, a certificate will be provided to Alter Trading Company with the following statement:

"*This certifies and verifies that the AEDA residue, Range Residue, MEC scrap and/or Explosive Contaminated property listed have been 100 percent properly inspected and to the best of our knowledge and belief, are inert and/or free of explosives or related material.*"

Figure 4-1. MEC Investigation Exclusion Zone

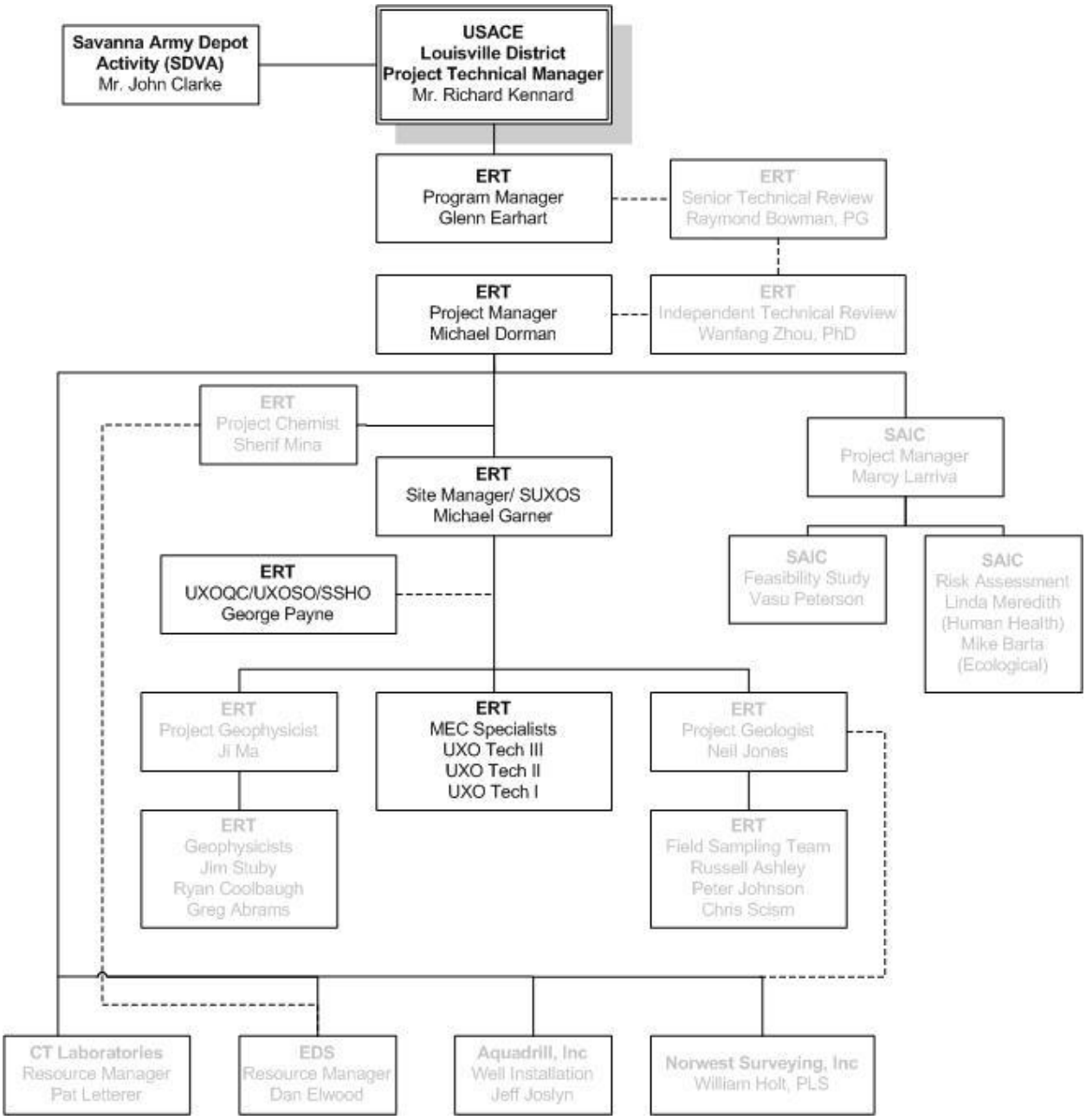


Figure 4-2. MEC Investigation Organizational Chart

Table 4-1. Minimum Safe Distance from Electro-Explosive Devices (EEDs)

AVERAGE OR PEAK TRANSMITTER POWER IN WATTS	MINIMUM DISTANCE TO TRANSMITTER IN METERS/FEET
0-30	30/98.4
31-50	50/164.1
51-100	110/360
101-250	160/525
251-500	230/755
501-1,000	305/1,000
1,001-3,000	480/1,575
3,001-5,000	610/2,001
5,001-20,000	915/3,002
20,001-50,000	1,530/5,020
50,001-100,000	3,050/10,007
100,001-400,000	6,100/20,014
400,001-1,600,000	12,200/40,028
1,600,001-6,400,000	24,400/80,056

Table 4-2. Minimum Safe Distances Between Mobile RF Transmitters and Electric Blasting Operations

Transmitter Power (Watts)	MF 1.6 to 3.4 MHz Industrial	HF 28 to 29.7 MHz Amateur	VHF 35 to 36 MHz 42 to 44 MHz 50 to 64 MHz	VHF 144 to 148 MHz 150.8 to 161.6 MHz	UHF 450 to 460 MHz Cellular Car Phones above 800 MHz
5	30	70	60	20	10
10	40	100	80	30	20
50	90	230	180	70	40
100	120	320	260	100	60
180	172	430	350	130	80

5.0 MEC HAZARDS AND SAFETY

In addition to the guidelines established in the HASP, the following general safety procedures will be followed during all MEC activities:

- A minimum of two UXO-qualified personnel will be present during all MEC intrusive operations so that one UXO personnel may act as a safety observer.
- During all MEC operations, only the minimum number of personnel required to safely perform the task will be allowed onsite. All others will evacuate to a pre-designated assembly point.
- If an unidentifiable MEC or toxic chemical munition is found, the SVDA BEC or Louisville District representative will be notified for directions or request EOD support.
- UXO personnel required for this project will include qualified UXO supervisors and technicians, all of who possess the relevant United States military EOD qualifications and experience. Personnel for this project have been selected from a pool of available qualified UXO technicians. All qualifications will be verified prior to any technicians entering the work area. All UXO personnel assigned to this project will meet the personnel training and experience requirements set forth in the Statement of Work (SOW) and DID MR-025.01 (USACE, 2002a).
- A Team Separation Distance (TSD) of 200 ft will be maintained, based on the MPM being a 155mm M107 HE projectile.

6.0 EXPLOSIVE MANAGEMENT PLAN

The purpose of this plan is to describe the procedures that will be used in the management, transportation, storage, and accounting of high explosives and initiators for use during MEC disposal operations at SVAD-050.

Procedures to manage the explosives for this project will be conducted in accordance with the following policies and federal, state, and local laws and regulations:

- Applicable sections of U.S. Department of Transportation (DOT), 49 CFR 100 to 199, Transportation;
- U.S. Bureau of Alcohol, Tobacco, and Firearms (ATF) Publication 5400.7, Alcohol, Tobacco, and Firearms - Explosives Law and Regulations (2000);
- USACE Engineer Manual (EM) 385-1-1, Safety and Health Requirements Manual (2003);
- DoD 6055.9-STD, DoD Ammunition and Explosives Safety Standards (1999);
- DOT regulations;
- Department of the Army (DA) PAM 385-64, Ammunition and Explosives Safety Standards (1999); and
- Army Regulation 190-11, Physical Security of Arms, Ammunition, and Explosives (DA 1998).
- Illinois Explosives Act, 225 ILCS 210, Public Act 86-364 (and all requirements for licenses, storage, inspection, and certifications as described in the Act).

6.1 Licenses/Permits

ERT will maintain the required licenses/permits to procure, store, and use explosives during the performance of MEC disposal operations at SVAD-050. Required licenses/permits will be posted and available for inspection at the ERT work site office (Building 1100). If required, Illinois State permits will be obtained. At a minimum, ERT will maintain copies of the following documents:

- ATF User of High Explosives license.
- Letter signed by an ERT official designating specific ERT personnel authorized to purchase, receive, access, or use explosives. Individual personnel may have one or more of these authorizations.
- Copies of ATF approval letters for personnel utilizing explosives.
- Copies of Illinois State Blaster's Licensing, if required.

6.2 Description and Estimated Quantity of Explosives

It is estimated that two Electric Blasting Caps (1.4B); and one 1-pound cast booster charge (1.1D) will be used during disposal operations for a single item. For venting operations, 32 gram Perforators (1.1D) may be substituted for cast boosters and up to 500 ft. of Detonation Cord (1.1D) may be used to link Perforators and or cast boosters.

6.3 Acquisition of Explosives

ERT has an ATF license (see Figure 6-1) and will obtain an Illinois Blaster's License if required in order to purchase and use explosives for disposal operations at SVAD-050. Explosives used

for disposal operations will be obtained from Austin Powder Company and will be ordered on an as-needed basis. Contact information for the Austin Powder Company is as follows:

Austin Powder Company
1119 Red Brick Road, Dixon, Illinois
Ph: (815) 284-8616.

6.4 Initial Receipt

A copy of the invoice(s) for the incoming donor materials will be kept in the on-site donor materials accountability file. Upon receipt of donor materials, an inventory will be conducted to ascertain:

- Correct type
- Serviceable condition

Upon receipt, a separate ERT Memorandum will be prepared, with the following information, and retained on-site.

- Date of acquisition
- Name or brand name of manufacturer
- Manufacturer's marks of identification
- Quantity
- Description
- Name, address, and license number of the persons from whom the explosive materials are received
- Items for which these explosives are being used to destroy or vent
- A statement that all explosives received were used the same day as received

6.4.1 Receipt

Ultimately the SUXOS will be responsible for the receipt and management of explosives used at this job site. The SUXOS will inventory, initiate, and maintain all documentation concerning the explosives upon receipt. Upon receipt, the type, quantity, and lot number of each explosive item will be checked against the manifest and recorded on the Explosives Usage Log (Attachment 3). Original explosive records will be archived in accordance with ATF regulations and requirements. ATF requires ERT to maintain explosive records for commercial purchases for 15 years. Copies of all records will be maintained onsite by the SUXOS and be available for inspection by authorized agencies.

6.4.2 Reconciling Discrepancies

The SUXOS will conduct a 100% inventory of the incoming explosives with the assistance of the UXOSO. The quantities annotated on the receipt documentation should match the quantities reflected in the inventory. If these quantities do not match, the Demolition Supervisor (a UXO Technician III) will contact the ERT Project Manager. The ERT Project Manager will contact the originator of the receipt documentation and reconcile any differences. ERT personnel will only sign for the actual quantity of material received. Receipt documentation will be changed to

reflect the proper quantities. Actual quantities will be properly annotated on the shipping documentation prior to ERT accepting delivery. These procedures will be conducted for each receipt of explosive materials.

6.5 Explosives Storage

ERT will not store explosives on the site for this operation. Explosives will be ordered only in quantities needed for demolition operations on the day they are delivered.

6.5.1 Physical Security

Explosives received on-site will be maintained in ATF-approved day boxes and will remain in the custody of the SUXOS and/or the UXOSO/UXOQCS. At no time will explosives be left unguarded.

6.5.2 Firefighting

A minimum of two serviceable Class ABC 20 LB fire extinguishers will be present when disposal operations are being conducted. One will be maintained in the ERT vehicle while transporting the explosives and one will be maintained at the demolition site. Local fire and medical will be contacted and made aware of the operation taking place.

6.6 Transportation of Explosives

Transportation of explosives and initiators to SVDA will be the responsibility of Austin Powder Company. They will have and maintain all permits and documentation required by federal, state, and local regulations regarding the transportation of explosives over the road to the point at which ERT will take custody of the explosives.

Once ERT has received the explosives they will be secured in the bed of the transport vehicle in appropriate storage containers protected by sandbag barriers. Initiators and explosives will not be transported in the bed of the vehicle. The initiators will be transported in a separate Blasting Cap Transport Container in the front cab of the vehicle.

Vehicles used for transportation of explosive materials will not be loaded beyond their rated capacity and the explosive materials will be secured to prevent shifting of load or dislodgment from the vehicle. All vehicles transporting explosive materials will display all placards, lettering, and/or numbering required by DOT and will have 10BC fire extinguishers on board.

Explosives will not be exposed to sparking metal during transportation of materials and all electric wiring will be completely protected and securely fastened to prevent short circuits; an inspection of wiring will be performed and a written record of such inspection will be kept on file. Persons employed in the transportation, handling, or other use of explosive materials will not smoke or carry on their persons or in the vehicle, matches, firearms, ammunition, or flame-producing devices.

Vehicles transporting explosive materials will not be left unattended. Only persons who are licensed users in Illinois will receive or handle explosives.

6.7 Inventory

The SUXOS will strictly control access to all explosives. The SUXOS will review all requests for explosives. Only sufficient explosives for the day's operations will be requested and issued.

Explosives will be inventoried when received. Only the amount of explosives ordered will be received. These quantities and lot numbers will be recorded on the Explosives Usage Log (Attachment 3).

6.8 Reporting Loss or Theft of Explosive Materials

If it is confirmed that explosives or initiators are missing, the SUXOS will notify the ERT Project Manager, the Louisville District, and the ERT Headquarters in Annapolis Junction, MD. ERT will notify ATF and immediately begin an investigation. The following notifications will be made:

- Contracting Officer via telephone within 1 hour of discovery
- Local Police: 911
- ATF: (410) 779-1700 (Baltimore Field Office)
- ERT Project Manager: 310-323-1401
- Contracting Officer in writing within 24 hours
- Appropriate local law enforcement authorities in writing within 24 hours

6.9 Disbursement of Explosives

The SUXOS will issue explosives to the Demolition Team Leader using the Explosives Usage Log (Attachment 3). These forms will be maintained in the site office.

The Demolition Team Leader receiving the explosives from the SUXOS will conduct a 100% inventory of the material. The quantities annotated on the Explosives Usage Log should match the quantities reflected in the inventory. If these quantities do not match, the Demolition Team Leader will bring this to the attention of the SUXOS.

6.10 Certification of Use

The Explosives Usage Log certifies that the explosives were received and expended as intended in the MEC disposal process. The Explosives Usage Log will be completely filled out for every demolition operation. The SUXOS will retain the originals in the project files.

6.11 Explosives not Expended

All explosive materials ordered and received will be used the same day. If there are any explosives left over, they will be taken to the Active Demolition Area and be destroyed.

6.12 Administration

ERT will obtain and retain a permanent file of all applicable demolition records, including permits, explosives recipes documentation, explosives usage records, training records, inspector reports, and any other applicable documentation.

DEPARTMENT OF THE TREASURY - BUREAU OF ALCOHOL, TOBACCO AND FIREARMS

LICENSE/PERMIT (18 U.S.C. CHAPTER 40, EXPLOSIVES)

In accordance with the provisions of Title XI, Organized Crime Control Act of 1970, and the regulations issued thereunder (27 CFR Part 555)you may engage in the activity specified in this license/permit within the limitations of Chapter 40, Title 18, United States Code and the regulations issued thereunder, until the expiration date shown. See "WARNING" and "NOTICES" on back.

DIRECT ATF CORRESPONDENCE TO
Christopher R. Reeves
Chief, Federal Explosives Licensing Center (FELC)
Bureau of Alcohol, Tobacco, Firearms and Explosives
244 Needy Road
Martinsburg, West Virginia 25401-9431
Telephone: 1-877-283-3352 Fax: 1-304-260-1141

LICENSE/ PERMIT NUMBER: 8-MD-027-33-9H-00254

EXPIRATION DATE: August 1, 2009

NAME
EARTH RESOURCES TECHNOLOGY INC

Premises Address CHANGES? You must notify the FELC at least 10 days before the move.
10810 GUILFORD ROAD SUITE 105
ANNAPOLIS JUNCTION, MD 20701-

TYPE OF LICENSE OR PERMIT
33-USER OF HIGH EXPLOSIVES

CHIEF, FEDERAL EXPLOSIVES LICENSING CENTER (FELC)
Christopher R. Reeves
Christopher R. Reeves

PURCHASING CERTIFICATION
I certify that this is a true copy of a license/permit issued to me to engage in the activity specified.

(SIGNATURE OF LICENSEE/PERMITTEE)

Mailing Address CHANGES? You must notify the FELC at least 10 days before the change.
EARTH RESOURCES TECHNOLOGY INC
10810 GUILFORD ROAD SUITE 105
ANNAPOLIS JUNCTION, MD 20701-

The licensee/permittee named herein shall use a reproduction of this license/permit to assist a transferor of explosives to verify the identity and status of the licensee/permittee as provided in 27 CFR Part 555. The signature on each reproduction must be an ORIGINAL signature.

ATF F 5400.14/5400.15, Part 1 (8/89)

DEPARTMENT OF JUSTICE

Bureau of Alcohol, Tobacco,
Firearms and Explosives

Martinsburg, WV 25405

August 11, 2009

Earth Resources Technology Inc
10810 Guilford Road Suite 105
Annapolis Junction, MD 20701

901090:CRR/KCU
5400
File Number: 8-MD-00254

Premises Address: 10810 Guilford Road Suite 105, Annapolis Junction, MD 20701

Dear Sir/Madam:

This letter acknowledges receipt of your timely application to renew your Federal explosives license/permit.

The Bureau of Alcohol, Tobacco, Firearms and Explosives (ATF) is not able to process your application prior to the expiration date of your license/permit. However, Federal law allows you to continue operations under your current license/permit until such time as ATF completes processing your application. See 5 U.S.C. § 558. This letter, or as explained below, a follow-up letter, will serve as your license/permit until we complete action on your renewal. It is referred to as a Letter of Authorization (LOA).

Since we have not completed processing your application, you may supply a copy of this letter to other licensees/permittees, e.g., your distributors, for the next six months (or until we complete action on your renewal, if that occurs in less than six months) as evidence of your licensed/permitted status. If we have not completed processing your application for renewal within six months of the date of this letter, we will send you another letter, which will also be valid for six months (or until we complete action on your renewal, if that occurs in less than six months). This is of course contingent upon your remaining entitled to continue operations under your current license/permit.

Please direct questions or concerns regarding this letter to Shannon Siviero at 877-283-3352.

Sincerely,

Christopher R. Reeves

Christopher R. Reeves
Chief, Federal Explosives Licensing Center

ATF web address: www.atf.gov

Figure 6-1. ERT ATF License

7.0 EXPLOSIVE SITING PLAN

The Explosives Siting Plan will be used by ERT to provide explosive safety criteria for planning and siting explosives operations at SVAD-050. This plan was prepared in accordance with DID OE-005-04.01 (USACE, 2002b) requirements.

7.1 Ordnance and Explosives Areas

All MEC investigations will take place within SVAD-050. Three different areas can be identified within SVAD-050 based on historic uses. These are the OD Area, the OB Area, and the other areas (e.g., roads). A description of each of these areas can be found in the primary Work Plan document.

7.2 Demolitions Areas

Live items identified by ERT UXO Teams, and with the concurrence of the USACE Safety Specialist, as too hazardous to move will be BIP after positive identification. Items identified by ERT UXO teams, and with the concurrence of the SUXOS and UXOSO, as safe to move will be consolidated and demilitarized between the existing berms within the OD area.

7.3 Acceptable Separation Distances

The Minimum Separation Distance (MSD) has been calculated by the U.S. Army Engineering and Support Center, Huntsville, Engineering Directorate, based on the MPM, a 155mm Projectile, M107 HE. The MFR is 2,577 ft for intentional detonations without engineering controls. Therefore an MSD of 2,577 ft is established and will be used as the EZ for the protection of non-essential project personnel and the public from blast overpressure and fragmentation hazards during demolition operations (Figure 4-1). In the event a MEC item that is suspected of having a greater maximum fragmentation distance is identified during the investigation, the BEC or appropriate Government representative will be notified and all distances will be adjusted, if necessary.

For all UXO work areas, the EZ will be established and identified by the UXOSO prior to the commencement of work. While MEC procedures are being conducted, only personnel essential to the operation will be allowed within the exclusion zone. At the discretion of the SUXOS, guards will be posted at points where the river enters the EZ to preclude river traffic within the EZ during demolition, or the EZ may be reduced through the use of engineering controls to move the slough outside of the EZ.

Control of access to the EZ will be established using existing locked gates and fences, where possible. Where locked gates and fences are not available, barricades will be established to prevent entry to the EZ.

If the above measures are not feasible, it may be necessary to place road guards, either to block traffic or to communicate to teams to cease UXO operations until nonessential persons have cleared the area.

For any demolition operations in the existing Open Demolition area, existing institutional controls will be used to control access to the EZ. If, because of water levels in the sloughs to the west or any other reason, the security of the EZ cannot be assured, then engineering controls will be used to reduce the EZ. These engineering controls will consist of sandbag enclosures

constructed in accordance with CEHNC-ED-CS-S-98-7 (USACE, 1998). In the event that BIP operations occur, the EZ may be reduced by the use of engineering controls with the approval of the UXOSO or authorized Government representative. The modified MSD for demolition operations using engineering controls is 220 feet. This MSD assumes the use of less than 1.25 pounds of donor charge and sand bag enclosure of demolitions, with an MPM item to be a 155mm M107 projectile.

7.4 Footprint Areas

7.4.1 Blow-in Place and Consolidated Shots

Prior to initiation of demolition operations, all nonessential personnel will be evacuated from inside the MSD. Prior to priming the demolition charges, all avenues of ingress will be physically blocked by guard personnel. Radio communications will be maintained between involved parties at all times. Avenues of ingress will not be opened without the express permission of the SUXOS. A constant state of vigilance will be maintained by all personnel to detect any intrusion into the EZ. The exclusion zone for BIP and transported, co-located demolitions is 2,577 feet based on an MPM of a 155mm Projectile, M107 (see Work Plan Appendix B, Map B-2). The EZ for BIP operations may be reduced to 220 feet if a Sandbag Enclosure is used in accordance with CEHNC-ED-CS-S-98-7 (USACE, 1998) and the donor charge does not exceed 1.25 pounds.

The UXO Tech III in charge of the MEC Sweep team will assign team members to specific demolition duties.

Destruction of UXO/MEC will be accomplished by detonation utilizing electrical firing systems to ensure maximum control and safety. Disposal by detonation will be conducted within approved procedures, regulations and guidelines.

7.4.2 Collection Points

MEC collection points will not be used as items will be either disposed of in-situ following BIP procedures, or left in-situ with engineering controls (sandbag cover and location marked) until the item can be transported and co-located for demolition at a later date. Demolition will occur on the last day of the work week, typically Friday.

7.5 Explosives Storage

ERT will not store explosives onsite. A licensed commercial vendor will deliver on an as-needed basis the type and quantity of explosives requested to support the day's disposal operation. The contracted vendor will be responsible for all permits and documentation required by federal, state, and local regulations regarding the transportation of explosives over the road to the point at which ERT will take custody of the explosives.

Explosives and initiators received on-site will be maintained in separate ATF-approved day boxes and will remain in the custody of the SUXOS and/or the UXOQCS. At no time will explosives be left unguarded.

7.6 Engineering Controls

In areas where acceptable fragmentation distances cannot be achieved, other methods of mitigation, such as sandbag barricades [in accordance with CEHNC-ED-CS-S-98-7 (USACE, 1998), will be employed to reduce the fragmentation hazard. If these methods of containment are determined to be impractical, the SUXOS will notify the SVDA BEC or Louisville District.

7.7 Site Maps

Figure 1-1 of the primary Work Plan document shows the geographic area of investigation. MEC investigation areas are shown in Figure 5-1 of the GIP and the EZ is shown in Figure 4-1 of this MEC Investigation Plan.

8.0 REFERENCES

ATF, 2000. *Explosives Law and Regulations*, Publication 5400.7

DA, 1969, *Explosive Ordnance Disposal Procedures*. TM 60A 1-1-31

DA, 1998, *Physical Security of Arms, Ammunition, and Explosives*, Army Regulation 190-11

DA, 1999, *Ammunition and Explosives Safety Standards Pamphlet*. PAM 385-64

DOD, 1997, *Defense Materiel Disposition Manual*, DOD 4160.21-M

DOD, 2008. *Ammunition and Explosives Safety*, DOD 6055.9-STD

ERT, 2009a, *Geophysical Investigation Plan for Remedial Investigaion/Feasibility Study (RI/FS) at Open Burn/Open Detonation Area (SVAD-050), Savanna Army Depot, Savanna, Illinois.*

ERT, 2009b, *Site-Specific Safety and Health Plan for Remedial Investigation/Feasibility Study (RI/FS) at Open Burn/Open Detonation Area (SVAD-050), Savanna Army Depot, Savanna, Illinois.*

USACE, 1998. *Use of Sandbags for Mitigation of Fragmentation and Blast Effects Due to Intentional Detonation of Munitions*, CEHNC-ED-CS-S-98-7

USACE, 2001, *Basic Safety Concepts and Considerations for Ordnance and Explosives Operations.* EP 385-1-95a

USACE, 2002a, *Personnel/Work Standards*, DID OE-025.01

USACE, 2002b, *Explosive Siteing Plan*, DID OE-005-04.01

USACE, 2003. *Engineer Manual (EM), Safety and Health Requirements Manual*, EM 385-1-1.

USACE, 2004, *Engineer Pamphlet (EP) Basic Safety Concepts and Considerations for Munitions and Explosives of Concern (MEC) Response Action Operations*, EP 385-1-95a.

USACE, 2007, *Geophysical Investigation Plan*, DID MR-005-05.01

US Department of Transportation, 2008. *Explosives Transportation* Regulation 49 CFR 100 – 199.

Attachment 1
MEC Accountability Log

MEC and MPPEH (1X and 3X) ACCOUNTABILITY INVENTORY

Date: _____							
RPT #	Item	Category*	Found (Grid/Date)	Location	Initial Disposition	Photo #	Final Disposition /Date

Notes

MEC-Munitions and Explosives of Concern-Specific categories of military munitions that may pose unique explosives safety risks; UXO, MPPEH, and MC

UXO – Unexploded ordnance – Ordnance fuzed, armed, or otherwise prepared for action and which has been fired or placed in such manner that constitutes a hazard.

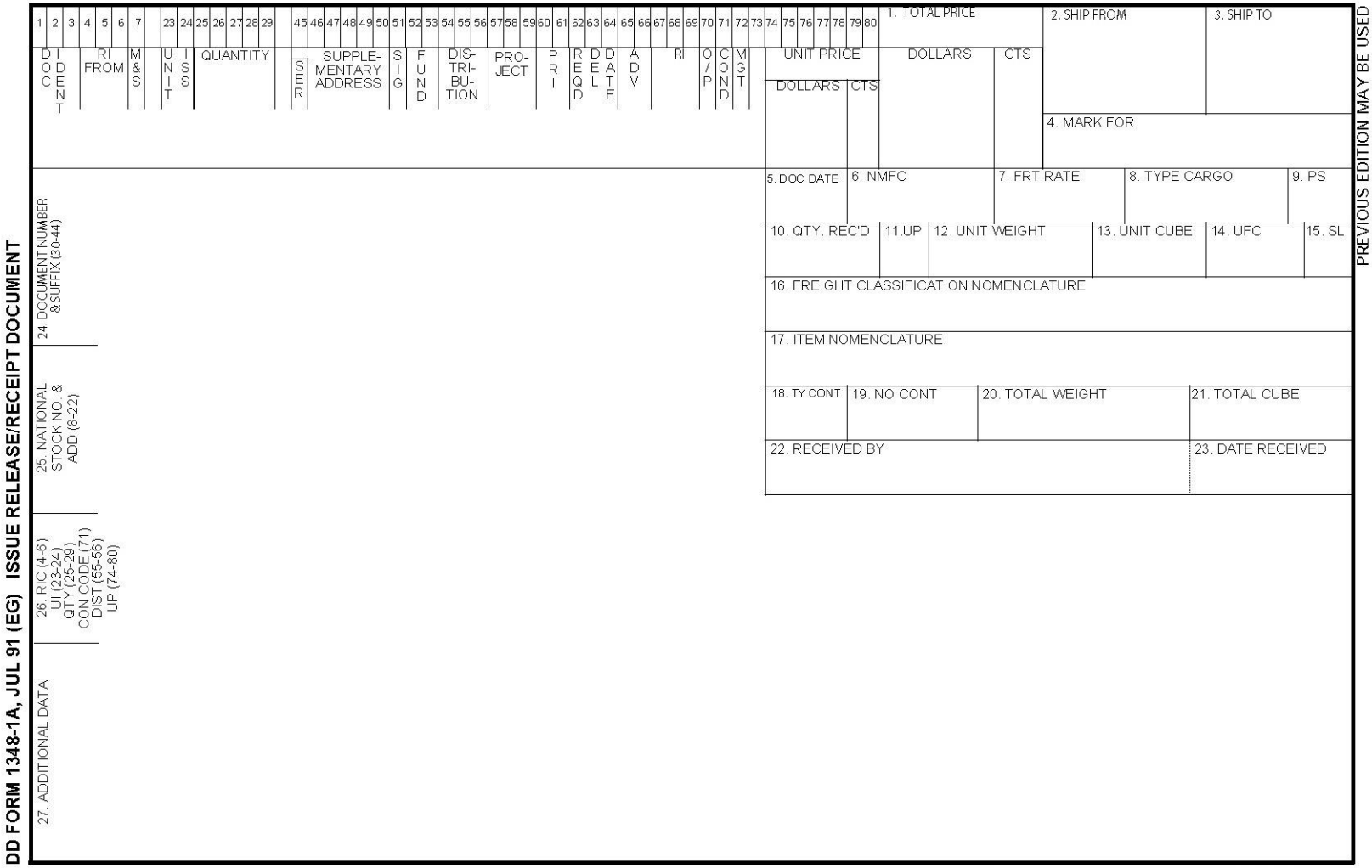
Material Potentially Presenting an Explosive Hazard (MPPEH).-Material potentially containing explosives or munitions (e.g., munitions containers and packaging material; munitions debris remaining after munitions use, demilitarization, or disposal; and range-related debris); or material potentially containing a high enough concentration of explosives such that the material presents an explosive hazard (e.g., equipment, drainage systems, holding tanks, piping, or ventilation ducts that were associated with munitions production, demilitarization or disposal operations). Excluded from MPPEH are munitions within DoD's established munitions management system and other hazardous items that may present explosion hazards (e.g., gasoline cans, compressed gas cylinders) that are not munitions and are not intended for use as munitions.

DMM – Discarded Military Munitions which was disposed of by abandonment. The munitions may or may not have been fuzed or armed, but was not employed as designed.

Inert – An item that has the same physical features of an ordnance item, but does not and never did contain energetic material.

MC - Munitions Constituents - Any materials originating from UXO, DMM, or other military munitions, including explosive and non-explosive materials, and emission, degradation, or breakdown elements of such ordnance or munitions.

Attachment 2
DD 1348-1


DD FORM 1348-1A, JUL 91 (EG) ISSUE RELEASE/RECEIPT DOCUMENT
PREVIOUS EDITION MAY BE USED
1 2 3 4 5 6 7 23 24 25 26 27 28 29 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80
DOC IDENT
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UNIT ISS
QUANTITY
SER
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FUND
DIS-TRI-BU-TION
PRO-JECT
PRI
REQD
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ADV
RI
O/P
COND
MGT
UNIT PRICE
DOLLARS CTS
1. TOTAL PRICE
DOLLARS
CTS
2. SHIP FROM
3. SHIP TO
4. MARK FOR
5. DOC DATE
6. NMFC
7. FRT RATE
8. TYPE CARGO
9. PS
10. QTY. REC'D
11.UP
12. UNIT WEIGHT
13. UNIT CUBE
14. UFC
15. SL
16. FREIGHT CLASSIFICATION NOMENCLATURE
17. ITEM NOMENCLATURE
18. TY CONT
19. NO CONT
20. TOTAL WEIGHT
21. TOTAL CUBE
22. RECEIVED BY
23. DATE RECEIVED
24. DOCUMENT NUMBER & SUFFIX (30-44)
25. NATIONAL STOCK NO. & ADD (8-22)
26. RIC (4-6)
UI (23-24)
QTY (25-29)
CON CODE (71)
DIST (55-56)
UP (74-80)
27. ADDITIONAL DATA

Attachment 3
Explosives Usage Log

EXPLOSIVES USAGE RECORD

Contract:		**Project Name:**	
Date:		**Work Area & Grid Numbers:**	
Team Number:		**Team Leader:**	
Explosives Issued		**Signature of Team Leader:**	
Item	**Quantity**	**Lot Number**	**Checker's Initials**
Explosives Expended		**Signature of Team Leader:**	
Item	**Quantity**	**Lot Number**	**Checker's Initials**
Explosives Returned		**Signature of SUXOS:**	
Item	**Quantity**	**Lot Number**	**Checker's Initials**

The signatures in each section of this document indicate that the items listed in that section were in fact issued, expended , or returned to storage and that the quantities listed were verified through a physical count.

APPENDIX D
SAMPLING AND ANALYSIS PLAN

FINAL
SAMPLING AND ANALYSIS PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT OPEN BURN/OPEN DETONATION AREA (SVAD-050), SAVANNA ARMY DEPOT SAVANNA, ILLINOIS

Prepared for

U.S Army Corps of Engineers
Louisville District

Contract Number: W912QR-08-D-0012
Delivery Order 0001

Prepared by

Earth Resources Technology, Inc.
4890 University Square, Suite 2A
Huntsville, Alabama 35816
(256) 217-8991

September 2009

FINAL
SAMPLING AND ANALYSIS PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT
OPEN BURN/OPEN DETONATION AREA (SVAD-050),
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APPROVED BY

9/1/09

Michael Dorman — Date
Project Manager

5/20/09

Glenn Earhart — Date
Program Manager

COMPLETION OF SENIOR TECHNICAL REVIEW

This document has been produced within the framework of the Earth Resources Technology, Inc. (ERT) and ISO Approved 9001:2000 quality management systems certification As such, a senior technical review, as defined in the project Quality Control Plan, has been conducted. This included review of the overall design addressed within the document, proposed or utilized technologies and alternatives and their applications with respect to project objectives, and Department of the Army policy and regulatory guidance and constraints

Raymond D. Bowman 12/19/08

Raymond Bowman, PG Date
Senior Technical Reviewer

COMPLETION OF INDEPENDENT TECHNICAL REVIEW

This document has been produced within the framework of ERT's total quality management system and ISO 9001:2000 certification. As such, an independent technical review, appropriate to the level of risk and complexity inherent in the project as defined in the Quality Control Plan for this project, has been conducted. This included review of assumptions (methods, procedures, and material used in analyses), alternatives evaluated; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets the project objectives. Comments and concerns resulting from review of the document have been addressed and corrected as necessary.

Wanfang Zhou 5/20/09

Dr. Wanfang Zhou Date
Independent Technical Reviewer

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
AOC	Area of Concern
ARAR	Applicable or Relevant and Appropriate Requirements
bgs	Below Ground Surface
BRAC	Base Realignment and Closure
CERCLA	Comprehensive Environmental Response, Compensation and Recovery Act
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COPC	Constituent of Potential Concern
COR	Contracting Officer's Representative
DAF	Dilution Attenuation Factor
DGM	Digital Geophysical Mapping
DO	Dissolved Oxygen
DOD	Department of Defense
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EM	Engineering Manual
EPA	United States Environmental Protection Agency
ER	Engineering Regulation
ERB	Equipment Rinsate Blank
ERT	Earth Resources Technology, Inc.
eV	electron volts
°F	degrees Fahrenheit
FB	Field Blank
FS	Feasibility Study
FSP	Field Sampling Plan
Ft.	Foot/Feet
GIWP	Geophysical Investigation Work Plan
HCl	Hydrochloric Acid
IDW	Investigative-Derived Wastes
L	Liter
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
µg	Microgram
µg/L	Micrograms per Liter
mg	Milligram
mg/L	Milligrams per Liter
min	Minute
mL	Milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MW	Monitoring Well
NELAP	National Environmental Laboratory Accreditation Program

NPL	National Priorities List
NTU	Nephelometric Turbidity Unit
OB	Open Burn
OD	Open Detonation
OE	Ordnance and Explosives
oz	Ounce
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PCB	Polychlorinated Biphenyl
PETN	Pentaerythritol Tetranitrate
PID	Photoionization Detector
PM	Project Manager
PQL	Practical Quantitation Limit
PQO	Project Quality Objective
ppm	Parts per Million
PRG	Preliminary Remediation Goal
PVC	Polyvinylchloride
QA	Quality Assurance
QAM	Quality Assurance Manual
QAO	Quality Assurance Objective
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCM	Quality Control Manager
QCP	Quality Control Plan
QL	Quantitation Limit
QSM	Quality Systems Manual
RBC	Risk-based Criteria
RCRA	Resource Conservation and Recovery Act
RDX	Cyclotrimethylenetrinitramine
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SEDD/ADR	Staged Electronic Data Deliverable/ Automated Data Review
SL	Screening Level
SOP	Standard Operating Procedure
SOW	Scope of Work
SSHO	Site Safety and Health Officer
SSHP	Site-specific Safety and Health Plan
SVOC	Semivolatile Organic Compound
SSHP	Site Safety and Health Plan
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
TAL	Target Analyte List
TB	Trip Blank

TBC	To be Considered
TBD	To be Determined
TCL	Target Compound List
TNT	Trinitrotoluene
TOC	Total Organic Carbon
TOX	Total Organic Halides
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
UXO	Unexploded Ordnance
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

1.0 INTRODUCTION

The Sampling and Analysis Plan (SAP) has been prepared by Earth Resources Technology, Inc. (ERT) to support environmental sampling and analysis activities associated with the remedial investigation (RI) and feasibility study (FS) for the Savanna Army Depot (SVDA) Open Burn/Open Detonation (OB/OD) Area, also referred to as SVAD–050 (see Figure 1-1). ERT has been contracted by the U.S. Army Corps of Engineers (USACE), Louisville District, to develop this SAP which outlines actions required to complete RI activities under the regulatory authority of the Comprehensive Environmental Response, Compensation and Recovery Act (CERCLA). The facility is currently being decommissioned under the Base Realignment and Closure (BRAC) Program. This document includes the Field Sampling Plan (FSP) and should be reviewed with the Quality Assurance Project Plan (QAPP) (provided as Appendix E of the RI/FS Work Plan). Specific activities addressed in this SAP include groundwater, surface water, sediment and soil sampling.

The SAP should be used in conjunction with the project Site-specific Safety and Health Plan (SSHP). The SAP was prepared in accordance with EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans (USACE, 2001).

1.1 Site Background and Description

SVDA is a military facility operated by the Department of the Army since 1918. Historically SVDA has been utilized for storage, assembly and demolition of various explosives, such as trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), and pentaerythritol tetranitrate (PETN), by detonation. SVAD-050 is comprised of approximately 7 acres and consists of three main focus areas, an OB site, an OD site, and other areas (roads). The OB pads were used to burn unstable propellants and the OD grounds were used to detonate obsolete projectiles and ordnance. The OB sites consisted of metal pans elevated above concrete pads by steel beams used to dispose of propellant. The propellants were destroyed by burning and the residual ashes were containerized on-site for disposal. The OB pads were not used except for emergencies and the most recent burn event occurred in 1985. The OD area consisted of two long earthen berms where pits were located, three pits per berm. Obsolete projectiles were destroyed energetically with a series of "shots." After the series of "shots" the surrounding area was searched for unexploded material. Materials that were not sufficiently destroyed were either collected and prepared for the next series of shots or detonated in place. The OD grounds were most recently used in the disposal of unexploded ordnance (UXO) generated during SVDA restoration activities. The Resource, Conservation and Recovery Act (RCRA) interim status allowed demilitarization operations to continue until October 2008. At this point, RI activities discussed will operate under the authority of the CERCLA process. For more details on the history and usage of the OB/OD area including site maps, see Section 1 of the Quality Control Plan (QCP).

1.2 Scope and Objectives

The primary objective of this RI/FS is to delineate the nature and extent of constituents of potential concern (COPC) at SVAD-050. The primary chemical parameters to be analyzed include target analyte list (TAL) metals, target compound list (TCL) volatile organic compounds (VOCs), TCL semi-volatile organic compounds (SVOCs), explosives (including nitroguanidine, nitrocellulose and nitroglycerine), nitrate/nitrite (aqueous samples only), cyanide,

polychlorinated biphenyls (PCBs), pesticides, and perchlorate (groundwater only). ERT will be performing data validation on sampling data. Details of the sampling and analysis activities are included in Section 3.

This SAP, provided as Appendix D of the RI/FS Work Plan, presents site-specific sampling plans and procedures, and site-specific analytical plans and procedures for SVAD-050. The objective is to provide concise justification for sample placement, the number of proposed samples per matrix, and the analytical program.

Other planning documents, including the QAPP, SSHP, Munitions and Explosives of Concern (MEC) Investigation Plan, and Geophysical Investigation Plan (GIP) are included as appendices to the project RI/FS Work Plan.

The overall RI/FS project objectives for SVAD-050 include the following:

- Evaluate the nature and extent of COPC from former open burning and open detonation activities at the site.
- Evaluate whether existing COPC exceed applicable data quality limits as listed in Attachment 1 of the QAPP, thereby warranting further evaluation (potential risk assessment and/or removal action).
- Assess potential ecological and/or human health risks from constituents of potential concern.

If potential risks are present, accumulate data sufficient to quantitatively delineate the impacted area and provide an estimate of the total volume of impacted media. This information will be essential in the development of a future FS.

1.3 Project Organization and Responsibilities

ERT will have the responsibility and authority to develop, modify, and implement the FSP. The organizational structure of this project illustrates the project management organization, describes the responsibilities and authorities of key personnel, identifies the responsibilities of personnel to assess and implement the requirements established by the FSP, and describes the reporting relationships and lines of communication and authority. All on-site personnel are responsible for complying with the requirements of the SAP. The ERT Project Manager (PM), ERT Site Manager, and the ERT Site Safety and Health Officer (SSHO) are responsible for on-site implementation of the plans and ensuring all work requirements are enforced. Figure 1-2 provides the organization of the field sampling team. These individuals are identified below:

- ERT Project Manager – Michael Dorman
- ERT Site Manager – Michael Garner
- ERT Field Sampling Team Leader – Neil Jones
- ERT Site Safety and Health Officer – George Payne

The roles and responsibilities of these individuals are presented throughout this document, the SSHP, and the Quality Control Plan.

1.4 Non-measurement Data Acquisition

Non-measurement data used in the completion of this project may include previous report results and well construction information from previous investigations conducted by the Louisville District. It is assumed that the Louisville District will provide previously collected data to ERT to include in the RI/FS Report for SVAD-050.

1.5 Potentially Applicable or Relevant and Appropriate Requirements

RI/FS activities at SVAD-050 are being performed under the Department of the Army Base Realignment and Closure (BRAC) program and are intended to investigate potential environmental impacts associated with former Department of Defense (DoD) activities. The site is listed on the U.S. EPA National Priorities List (NPL); therefore, the RI/FS strictly adheres to the CERCLA process.

Under BRAC, the USACE performs environmental investigation activities consistent with the U.S. EPA under the CERCLA/Superfund Amendments and Reauthorization Act (SARA) program. As such, screening levels that will be used in assessing environmental conditions at SVAD-050 will be U.S. EPA Regional Screening Levels and any other applicable screening levels, as listed in Attachment 1 of the QAPP. These are discussed in more detail in the QAPP. Location-specific Applicable or Relevant and Appropriate Requirements (ARARs) and To Be Considered (TBC) criteria are restrictions placed on the concentrations of hazardous substances or the conduct of activities in specific locations or on locations with specific characteristics, for example, wetlands. Action-specific ARARs and TBC criteria are restrictions and guidelines placed on specific activities undertaken at project sites. For the sampling and analytical program addressed in the FSP, action-specific ARARs are limited to those regulating disposition of investigative-derived wastes (IDW).

1.5.1 Soil

Federal and State technical guidance documents may be relevant and appropriate for the evaluation of soil constituents reported at SVAD-050. U.S. EPA Regional Screening Levels (RSLs) and Illinois Tiered Approach to Corrective action Objectives (TACO) Tier 1 Soil Remediation Objectives for Residential Soil will be used as the primary guidance for evaluating constituents of potential concern. Other published guidance, applicable to specific media or currently in draft form, will be incorporated, as appropriate. Additional details are provided in the QAPP (Appendix D of the Work Plan). This guidance is intended to be used to screen out areas of sites, exposure pathways, or chemicals of concern from further consideration, assuming certain conditions are present, or to determine that further investigation is warranted at a site. Generally, when evaluating sample results, data which falls below screening levels may be eliminated from further assessment consideration. Areas above the screening levels generally warrant further evaluation. The presented screening levels are not intended to be used to represent the cleanup goals for a site, and soil concentrations which exceed screening levels should not be habitually considered to represent intrinsic risk.

A dilution attenuation factor (DAF) of 1 (negligible) will be used for the RI. The DAF is used to account for concentration reductions through natural attenuation processes including adsorption

onto soil, chemical transformations, degradation, and dilution within unaffected groundwater sources.

1.5.2 Groundwater

Federal and State technical guidance documents may be relevant and appropriate for the evaluation of groundwater constituents reported at SVAD-050. U.S. EPA RSLs for Tapwater, U.S. EPA Maximum Contaminant Limits (MCLs), and IEPA Groundwater Quality Standards (currently undergoing revisions) will be used for evaluating constituents of potential concern. This guidance is intended to be used to screen out areas of sites, exposure pathways, or chemicals of concern from further consideration, assuming certain conditions are present, or to determine that further investigation is warranted at a site. Generally, when evaluating sample results, data which falls below screening levels may be eliminated from further assessment consideration. Areas above the screening levels generally warrant further evaluation.

1.5.3 Surface Water

Federal and State technical guidance documents may be relevant and appropriate for the evaluation of surface water constituents reported at SVAD-050. Illinois Surface Water Standards for human health (or Illinois' surface water human health and aquatic life criteria for chemicals not listed in the SW Quality Standards) and U.S. EPA's RSLs for Tapwater will be used for evaluating constituents of potential concern. This guidance is intended to be used to screen out areas of sites, exposure pathways, or chemicals of concern from further consideration, assuming certain conditions are present, or to determine that further investigation is warranted at a site. Generally, when evaluating sample results, data which falls below criteria levels may be eliminated from further assessment consideration. Areas above the screening levels generally warrant further evaluation.

1.5.4 Sediment

Federal and State technical guidance documents may be relevant and appropriate for the evaluation of sediment constituents reported at SVAD-050. U.S. EPA's National Sediment Quality Screening values, Illinois Tiered Approach for Evaluation and Remediation of Petroleum Releases to Sediments, Illinois Sieved Stream Sediment Data, and U.S. EPA RSLs for Residential Soil will be used for evaluating constituents of potential concern. This guidance is intended to be used to screen out areas of sites, exposure pathways, or chemicals of concern from further consideration, assuming certain conditions are present, or to determine that further investigation is warranted at a site. Generally, when evaluating sample results, data which falls below criteria levels may be eliminated from further assessment consideration. Areas above the screening levels generally warrant further evaluation.

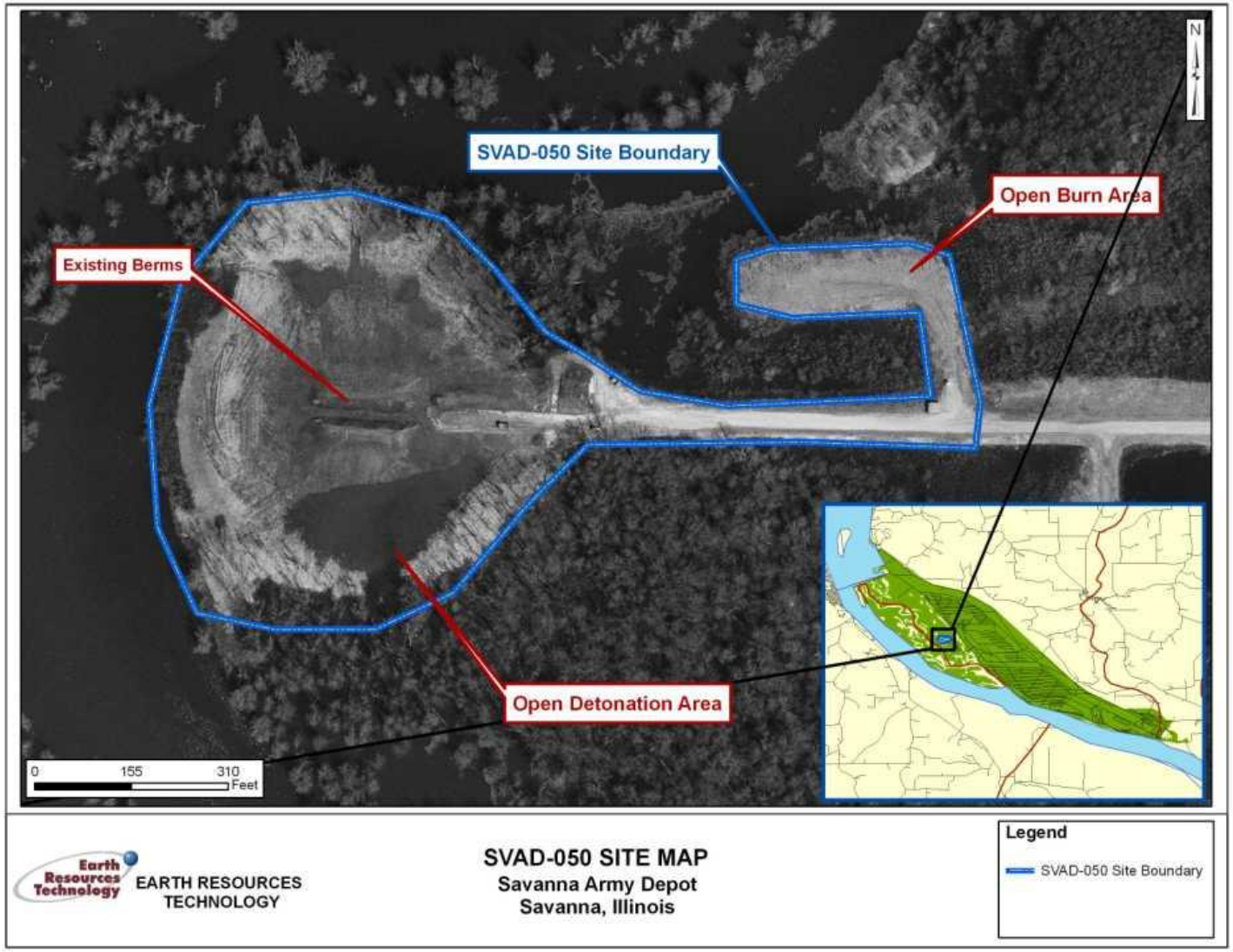

Figure 1-1. SVAD-050 Site Map

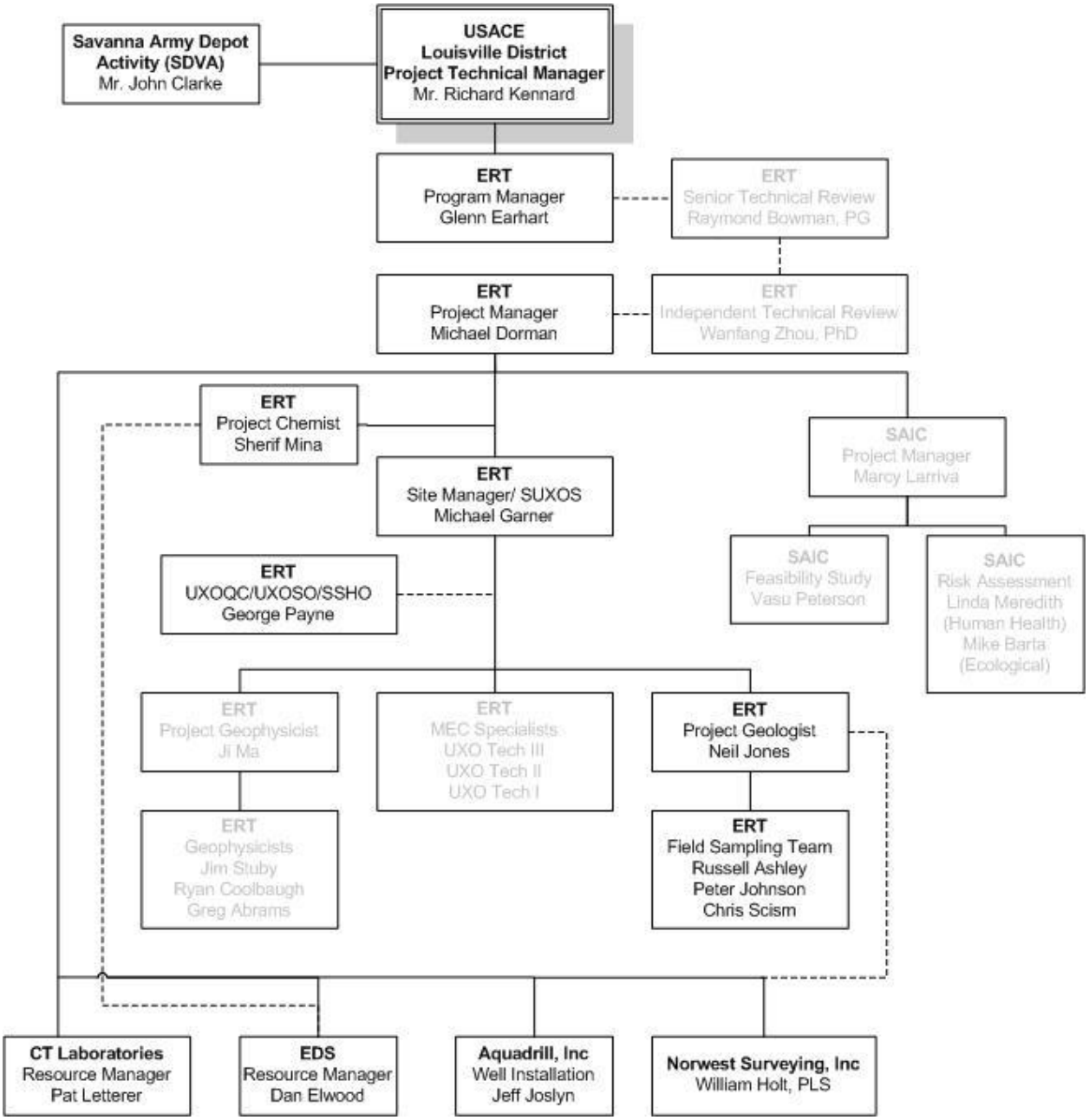

Figure 1-2. Field Sampling Team Organization

2.0 PRE-SAMPLING ACTIVITIES

Prior to sample collection, pre-sampling activities described in the following subsections will be performed. These activities include conducting meetings, as appropriate, with stakeholders so they are aware of the possible impacts to site-use, municipal and private utility checks, vegetation clearance, land surveys, munitions and explosives of concern (MEC) sweeps and interim cleanup, geophysical investigations and any other site preparation.

It is unknown if underground utilities are present in areas of potential intrusive activity. As per the Illinois Underground Utilities Facilities Damage Prevention Act, Illinois One Call will be contacted to perform utility location and mark outs for the property. ERT reserves the right to conduct further geophysical utility clearance as warranted.

2.1 Vegetation Clearance

Brush clearance may be required to access certain sample locations throughout SVAD-050. No more than 7 acres are anticipated to be cleared in order to facilitate MEC surface clearance, digital geophysical mapping (DGM) investigations and environmental sampling. It is not the intention to clear cut the entire acreage of SVAD-050. A more detailed description of the vegetation clearance process is provided in the MEC Investigation Plan provided as Appendix C to the RI/FS Work Plan.

Large debris and saplings (less than 4 inches in diameter) may be encountered during site preparation work. Any such material that inhibits MEC clearance, geophysical investigations, or access to a proposed sample location will be removed and piled on-site. An area local to the clearing efforts for the stockpiling of large vegetation and debris will be selected and agreed upon with representatives of the USACE-Louisville and the installation.

Due to known MEC within SVAD-050, all personnel will be required to be escorted by a UXO technician during all intrusive site activities associated with the RI or in areas that have not been surface cleared of MEC. Due to the necessity of clearing vegetation prior to conducting the MEC surface clearance, all vegetative clearing activities will be conducted by UXO technicians.

Areas where vegetation clearance is necessary will be clearly marked prior to clearance. These areas will be pre-determined by a site visit and boundaries will be marked utilizing stakes, construction tape/ribbon, marking paint, or similar. These limits are not intended to be professionally surveyed.

Vegetation clearing will be conducted in teams consisting of no less than two members. One member of the team will act as a scout and be required to walk the area intended to be cleared. The job of this individual will be to identify and mark large MEC ahead of machine clearance. This individual will be equipped with a machete, hatchet, chain-saw, or similar and will remove saplings/vegetation unable to be cleared by machine. Observed MEC will be marked so as to be easily visible, and left in place. UXO professionals conducting vegetative clearance will not handle, move or otherwise come in contact with MEC during this activity.

The second member of the vegetation clearance team will operate a bush hog or similar clearing apparatus, if necessary. The cutting blade will be set to a height of no less than 6 inches from the ground surface in an effort to minimally disrupt potential MEC. The operator and bush hog should follow a safe distance (minimum of 15 feet) behind the lead scout and never deviate from the path established by the scout.

2.2 Land Survey

A professional land survey will be conducted to establish a grid system utilized for MEC surface clearance, DGM investigation and field screening activities. The proposed grid system will cover the entire SVAD-050 and establish intersection nodes every 100 feet (ft) (50-foot grid in the OB area and the center of the OD area). Figure 2-1 presents the proposed grid system. At all times while on-site, land surveyors will be escorted by UXO technicians. Survey points will be clearly marked and labeled in the field. A certified land survey map of the grid system will be provided in the RI/FS Report.

2.3 MEC Surface Clearance

Prior to sampling events related to the RI activities, a MEC survey and clearance will be conducted for areas of proposed DGM surveys and environmental sampling. The MEC Investigation Plan details activities and safety hazards specific to the task and is presented as Appendix E to the RI/FS Work Plan.

2.4 Geophysical Investigations

Geophysical investigations will be conducted across various sections of SVAD-050 prior to sampling activities. The GIP details activity associated with this task and is presented as Appendix D to the Work Plan. Based upon the analysis of the geophysical investigation, six test pit excavations will be executed. As described in the GIP, criteria for selecting test pit excavation locations will be based on factors such as high concentrations of geophysical anomalies. A minimum of one test pit will be excavated at each of the OD and OB areas. Within each excavated test pit, a minimum of two soil samples, surface and subsurface, will be collected for laboratory analysis.

3.0 FIELD SAMPLING PLAN

This FSP has been prepared to ensure: (1) the project quality objectives (PQOs) specific to the RI are met, (2) the field sampling protocols are documented and reviewed in a consistent manner, and (3) the data collected are scientifically valid and defensible. ERT has prepared the FSP to define the standard operating procedures (SOPs) to be used during all sampling and analysis activities.

It is the purpose of the RI to delineate the nature and extent of COPCs at SVAD-050 and if environmental impacts are present, accumulate sufficient data to reasonably estimate the potential associated risks. If risks are determined, it is the objective of this sampling plan to provide adequate information capable of developing an accurate estimate of the amount of impacted media.

3.1 Proposed Sample Locations

The proposed Phase I soil, groundwater and Phase II surface water/sediment sample locations are summarized on Figures 3-1, 3-2, and 3-3 and Table 3-1. Table 3-1 includes the following information:

- Sample Location Designation
- Targeted Sample Collection Depth(s)
- Proposed Analysis
- Rationale for Sample Location

Sampling protocols are provided in the project QAPP. Proposed Phase I soil sampling consists of laboratory analysis for a minimum of two soil samples (one surface and one subsurface) collected from each test pit and surface soil field screening samples. Surface soil field screening will be conducted at 105 locations that include 88 systematic sample locations (based on grid nodes as shown in Figure 3-1) and 17 additional biased sample locations based on field observations. It is the intent of these proposed sampling locations to characterize and delineate potential impacts inherent to SVAD-050. After a review of the DGM investigations, field screening soil sample results and Phase I soil sample results, a second phase of surface and subsurface soil sampling will be conducted. Phase II soil sampling will consist of surface and subsurface samples collected for laboratory analysis from 25 locations. The Phase II sampling will also include groundwater sampling of the new monitoring well, surface water sampling and sediment sampling. Each sampling type is described in detail in the following sections of the FSP.

One groundwater well will be installed and sampled in the center of the berms of the OD area (see Figure 3-2). The results of this groundwater sample will be assessed with groundwater data generated from quarterly sampling events at eight wells within SVAD-050, to evaluate potential impacts to groundwater. If the results of the groundwater sample in the new well are sufficiently different than the results from the nine existing RCRA groundwater monitoring wells around SVAD-050, additional sampling may be recommended by the Army. The current RCRA groundwater monitoring, per the RCRA Closure Approval Letter, includes analyses of the following:

- Total and dissolved TAL Metals
- Total organic carbon (TOC)
- Total organic halides (TOX)
- Nitrate/nitrite
- Total cyanide
- Explosives (including nitroguandaine nitrocellulose, and nitroglycerine)
- Volatile organic compounds (VOCs)

Perchlorate analysis will be conducted during the fourth quarter 2009 groundwater sampling event and dissolved metals not be analyzed for the third and fourth quarter 2009 groundwater sampling events.

Sediment samples will be collected from a pond within the OD area and along the banks of open waterways adjacent to SVAD-050 (see Figure 3-3). The results of this sampling will be used to assess potential ecological impacts from previous site activities.

Surface water samples will be collected from a pond within the OD area and open waterways adjacent to SVAD-050 (see Figure 3-3). The results of this sampling will be used to determine potential ecological impacts from previous site activities.

3.2 Summary of Analytical Program

Samples will be submitted for full suite analysis to include TCL VOCs, TCL SVOCs, TAL metals, pesticides, polychlorinated biphenyls (PCB), perchlorate (groundwater only), cyanide, and explosives analysis as presented in the QAPP. Project-specific target compound and analyte lists are also available in the project QAPP. Table 3-1 summarizes the proposed number of samples during each phase of the sampling effort. Table 3-2 lists the analytical parameters to be used and 3-3 summarizes the frequency and type of quality control samples.

IDW classification sampling will be conducted to characterize soil, liquids and/or groundwater for the purpose of proper off-site disposal. Analysis will consist of only parameters requested by waste disposal vendors, the results of which will not be validated.

Detailed sampling protocols and the analytical program are presented in the QAPP. The project QAPP presents the data quality objectives (DQOs), required quality assurance (QA) and quality control (QC) samples, and rationale for collection

3.3 Field Sampling Procedures

ERT will be conducting the RI field activities at the SVAD-050 site. ERT will adhere to the protocols set forth in the approved RI/FS Work Plan. The sampling procedures will be conducted in accordance with established technical guidelines, methods, policies and standard operating procedures (SOPs). These SOPs are listed in Table 20 of the QAPP and provided in Attachment 3 of the QAPP. The subsections below present an overview of the sampling program procedures.

The potential exists for MEC to be encountered during RI activities. The environmental sampling team will be non-UXO-qualified personnel and will be escorted by a UXO Technician II or above during intrusive sampling activities or in areas that have not been surface cleared for MEC. The UXO Technician will conduct anomaly avoidance procedures to prevent anomalies from being excavated during soil sampling.

Anomaly avoidance procedures consist of a UXO Technician II or above conducting a visual surface sweep of the ground around the area proposed for soil sampling. Once cleared of surface hazards, the UXO Technician will use a magnetometer to sweep and clear the area of subsurface anomalies prior to allowing the environmental sampler access to the sample location.

3.4 Soil Sampling

In order to define the nature and extent of COPCs and delineate potential impacts, surface and subsurface soil samples will be collected from various sampling locations across the site. Soil sampling will be conducted in a two-phased sampling approach. Phase I soil sampling will consist of roughly 105 field screening soil samples and 12 soil samples collected for laboratory analysis from proposed test pit locations. At the conclusion of Phase I sampling, the results (both field screen and laboratory analytics) will be reviewed. Based upon this data and an evaluation of geophysical investigation results, a maximum of 25 sample locations will be selected for Phase II sampling, as areas of further investigation. Two soil samples will be collected from each Phase II sample location for laboratory analysis. One sample, a surface soil sample, will be collected from 0-6 inches below ground surface (bgs). The second soil sample will be collected from the interval of most likely impact. This interval will be selected based upon field observations and information obtained during the DGM investigation. This sampling approach will provide sufficient information for all analytical parameters across the OB/OD area to support future conclusions regarding site characterization. Figure 3-1 shows the sample locations to be utilized during Phase I surface soil screening sampling.

3.4.1 Phase I Soil Sampling Methods

Phase I sampling consists of field screen sampling on a pre-established grid system and samples collected from proposed test pit excavations. The sample methods for each are described below.

3.4.1.1 Systematic Field Screen Sampling

A systematic sampling approach has been designed for field screen soil sampling, which targets surface soil in the entire SVAD-050 area (see Figure 3-1). The intent of a systematic sampling approach is statistically and functionally similar to simple random sampling. The systematic sampling grid was developed to investigate potential impacts across SVAD-050. The objective of this sampling approach is to provide every component within the sample population with a known and equal probability of selection and assumes that the sample population is categorically homogeneous. The systematic field screening process will define whether the sample population is homogeneous or not.

Systematic sampling will be performed on a grid which extends across SVAD-050. Sample locations will be located on 50 ft or 100 ft centers, with the exception of where sample locations encounter former structures that are unable to be penetrated. Systematic samples originally placed in such described locations will be offset a minimal distance to remove the obstruction

from the sample location. A total of 88 systematic field screening sample locations, based on grid nodes, shown in Figure 3-1 and 17 additional biased field screening samples will be collected based on field observations during Phase I. The field screening samples will be collected as grab samples. These surface soil samples are to be analyzed for the detection of explosives, using Dropex® explosives detection kits.

Dropex® is a liquid–based detection system for the identification of Group A and B explosives through a series of sequential reactions. The Dropex® kits will be used to provide a rapid environmental screen of surface soil samples. Nitroaromatics, nitrate esters and nitramines are determined by a series of reactions which differentiates between the concentrations and sets of compounds within the media tested.

3.4.1.2 Test Pit Excavation Sampling

Based upon a review of the DGM Investigation results, six test pit locations will be selected. The process for selecting test pit locations is described in detail within the GIP provided as RI/FS Work Plan Appendix B. A minimum of one test pit will be excavated in each of the OD and OB areas. Each sample obtained from test pit locations will be laboratory analyzed for TCL VOCs, TCL SVOCs, TAL Metals, explosives, nitroguanidine, nitrocellulose, cyanide, PCBs, and pesticides.

Upon arriving at the test pit location, a discrete surface soil sample will be collected. Discrete/grab soil samples will be collected by retrieving a volume for analysis using a clean stainless steel, aluminum, or mild steel scoop, trowel, spoon, or bucket auger and placing the soil into the sample container. All parameters except VOCs and total organic halides will be homogenized. VOC sampling will be conducted using U.S. EPA Method 5035. Terra Core® samplers will be used to collect 5 grams of soil for low level VOC and total halide analysis.

After the entire extent of a test pit has been excavated and field logged with a logbook and camera, a subsurface sample location will be selected that represents the most likely impacted area. Sample locations will be determined by field personnel based upon visual and olfactory cues, Photoionization Detector (PID) readings and groundwater levels. If there are no observed potential impacts, the subsurface sample will be taken from the bottom of the excavation. In most cases, it is assumed that the discrete subsurface sample will be taken from the bottom of the test pit. It is not the intention of this investigation to chase anomalies that are uncovered during test pit excavations. If such anomalies are present, it may be necessary to obtain the subsurface sample from a test pit excavation sidewall.

3.4.2 Phase II Soil Sampling Methods

Biased sampling has been included as part of the two-phase soil sampling approach and is designed to target historic operations in areas which may constitute higher potential for impacts. Biased sampling is statistically significant when used in conjunction with a systematic sampling approach. A biased sample is one that is not representative of the population from which it was obtained.

At least 25 biased soil sampling locations will be selected for Phase II sampling based on the results of the geophysical investigation, test pit excavations, Phase I field screening results and

historical data review. The Army will prepare a technical memorandum summarizing these results and presenting the proposed Phase II soil sampling locations, which will be submitted to U.S. EPA Region 5 and IEPA for their review and concurrence, prior to initiating the Phase II efforts.

One surface sample and one subsurface sample will be collected for laboratory analysis from each biased sample location. For the purpose of this RI, surface samples will be considered and collected from 0-6 inches bgs. Subsurface samples will be considered those samples collected at some point below 6 inches bgs. Each sample will be analyzed for TAL metals, explosives, and any other parameter from related Phase I samples exceeding screening levels. Of the 25 soil sampling locations (50 total samples), four samples may be selected for waste characterization, using the Toxicity Characteristics Leaching Procedure (TCLP).

A hand auger (or similar) will be used to obtain subsurface soil samples. The hand auger will be advanced to the water table, or to the maximum depth of suspected impact based on a review of the historical information and field observations. If evidence of grossly impacted soil is encountered, the auger will be advanced to a depth at which soil no longer exhibits such impacts. Geologic descriptions of the soil and field screening results will be recorded in field logs.

Soil samples from each auger core will be screened using a PID to detect possible organic vapors. Organic vapor screening will be performed by making a small slice in the soil column with a clean knife or sampling tool, inserting the PID probe and pushing the slice closed, and monitoring the soil for approximately 5 to 10 seconds. This procedure will be repeated at intervals along the soil column at the field geologist's discretion.

The samples will be examined for staining, discoloration, odors, and debris indicative of contamination (ash, metal fragments, wood chips, cinders, petroleum staining, etc.). Samples for laboratory analysis will be collected from the six-inch interval most likely to be contaminated, based on PID readings, discoloration, staining, and the field geologist's judgment (field conditions may require a section longer than six inches to make sufficient sample; however this decision will be field-based). The samples will be collected by cutting the soil in two places with either disposable plastic trowels or a decontaminated steel, stainless steel, or aluminum trowel, spoon, or knife and homogenized in either a disposable plastic container or a decontaminated stainless steel pan before being placed in the sample bottles. VOC and total organic halides samples will go directly into the sample containers without homogenization. Samplers will wear phthalate-free gloves such as nitrile (no latex will be used) and will avoid contact of the gloves with the sample. Only clean metal instruments or dedicated disposable instruments will be allowed to touch the sample. If there is insufficient soil volume in the auger core, then this will be made up by attempting a second auger core at the same depth, or by using the next immediate sample interval above or below this depth, if appropriate. If there is no recovery, then the sample from this depth will be skipped, and auguring will progress to the next depth interval.

Soil samples collected from each auger hole will be screened with a PID and be inspected for indications of contamination (e.g., staining, odors, etc.). At a minimum, two samples from each probe hole will be selected for analysis. If there are no indications of contamination in the probe

hole, the sample closest to the water table/depth of potential concern and the sample located from zero to 6" bgs will be selected for analysis.

Grab soil/solid samples will be collected from the material or interval in question by retrieving a volume for analysis using a clean stainless steel, aluminum, plastic disposable or mild steel scoop, trowel, spoon, or bucket auger and placing the soil in a clean stainless steel pan or an inert disposable dish for homogenization before inserting into the sample container. All parameters except VOCs and total organic halides will be homogenized. Samples for VOC analysis and total organic halides will be placed directly into the sample container.

3.5 Sediment Sampling

Sediment samples will be collected and laboratory analyzed to determine potential impacts to human and ecologically sensitive receptors. Ten surface sediment samples (0-6 inches bgs) will be collected; two from within the pond located in the OD area and eight from sediment deposits along the open waterway bordering SVAD-050. In addition, five subsurface sediment samples will be taken, from a depth of 2 to 3 ft bgs, to define the potential vertical extent of any contamination. The proposed sample locations are presented in Figure 3-3. For the purpose of the RI, sediment will be considered soils present below the mean water level of any static or open water body that is saturated greater than 50% of the year. This definition is intended to exclude soils which lie at the base of puddles, drainage ditches, swales and other similar formations.

Discrete surface sediment samples will be collected. Surface sediments for the purpose of the RI, are considered sediments from 0-6 inches bgs. Discrete/grab soil samples will be collected by retrieving a volume for analysis using a clean stainless steel, aluminum, or mild steel auger, or disposable corer and placing the soil into the sample container. All parameters except VOCs and total organic halides will be homogenized. Samples for VOC analysis and total organic halides will be placed directly into the sample container. Sediment samples will be laboratory analyzed for TCL VOCs, TCL SVOCs, TAL metals, explosives, nitroguanadine, nitrocellulose, cyanide, pesticides, and PCBs.

3.6 Surface Water Sampling

Surface water samples will be collected and laboratory analyzed to determine potential impacts to human and ecologically sensitive receptors. Five surface water samples will be collected; one from within the pond located in the OD area and four from the open waterway bordering SVAD-050. The proposed sample locations are presented in Figure 3-3.

Where possible, grab surface water samples will be obtained by filling appropriate jar ware from along the shores of the proposed water bodies. If conditions do not permit this type of sampling, a grab sample of the surface water will be obtained by attaching a laboratory sterilized jar to a telescoping pole. The apparatus will be extended towards the center of the water body and water allowed to enter by submerging the jar. The jar will be retrieved when full and first used to fill three 40-ml glass screw cap volatile organic analysis (VOA) vials, each containing 1:1 HCl and a Teflon-faced silicone septum lid. The 40-ml glass VOA vials will be pre-cleaned, prepared, and certified by the laboratory. No head space or air bubbles will be left in the sample vials.

After the VOA vials have been properly filled and sealed, the process will be repeated as necessary to obtain adequate water volume to fill the remaining jar ware. Surface water samples will be analyzed for TCL VOCs, TCL SVOCs, TAL metals, explosives, nitroguanidine, nitrocellulose, nitrate/nitrite, cyanide, PCBs and pesticides.

3.7 Groundwater Sampling and Analysis

As a function of the RI, groundwater at the site will be analyzed and evaluated for potential impacts. The RCRA Quarterly Groundwater Monitoring Program at the OB/OD units began in 1996. Previous groundwater sampling data obtained from the quarterly sampling events are available and will be screened against the U.S. EPA MCLs and state groundwater quality standards and draft standards. In addition, one new groundwater monitoring well (MW-11) will be installed in the center of the OD area, between the two existing berms and sampled during the RI field activities. The additional well is located in the center of the OD area, the area with the greatest potential impact from past operations. The existing RCRA monitoring wells are located around the border of the OD site, and have been shown to be influenced with the height of the river (and potential mixing).

3.7.1 Monitoring Well Installation

One new groundwater monitoring well will be installed in the center of the OD area, between the two existing berms. The groundwater monitoring well will be installed utilizing a CME-55 drill rig, Geoprobe® 6620DT or similar, and constructed of threaded two-inch-diameter polyvinylchloride (PVC) well casing and 10-slot well screen. Clean silica sand, Morie No. 1, or equivalent, will be placed in the annular space around the well to a minimum of one foot above the top of the well screen, two feet being optimal. Solid PVC riser, attached to the well screen, will extend to grade or above if the well is a stick-up. For a two-inch diameter well, the annular space for the filter pack should be between 2 and 4 inches thick. (The 4 ¼" inside diameter hollow stem augers will have to be retracted as the filter pack is installed to yield the required annular space.) A two-foot thick bentonite seal will then be placed above the sand pack and moistened with potable water for a minimum of 15 minutes before backfilling the remaining space with a cement-bentonite grout. If warranted by depth, filling will be completed using a tremie pipe placed below the surface of the grout. A stick-up protective casing with a locking well cap will then be installed and a measuring point marked on each PVC well riser. Well construction diagrams will be prepared for the newly installed well.

Based on a review of the existing water table, existing groundwater monitoring well construction details and COPC, it is proposed that the new monitoring well be installed to an approximate depth of 20 ft bgs. The screened interval will extend between 20 ft bgs and 10 ft bgs. A PVC riser will extend from the top of the screen to above the ground surface.

All soil cuttings from the installation of MW-11 will be handled as IDW (see section 7.0 of WP).

3.7.2 Monitoring Well Development

Following installation, the groundwater monitoring well will be developed, using a two-inch diameter submersible pump (or equivalent) until the water is reasonably free of turbidity and field readings (pH, conductivity, temperature, and dissolved oxygen (DO)) sufficiently stabilize to within +10% for two consecutive readings. Five nephelometric turbidity units (NTU) will be

used as the target value for clean water. However, if 5 NTU is not achievable, sampling will be conducted only after the turbidity readings become stabilized to within 10% for two consecutive readings. The wells will be developed aggressively to remove fines from the formation and sand pack. The wells will be allowed to equilibrate for 14 days prior to sampling. The volume of water removed, the well development time, and field instrument readings will be recorded in the logbook. Well development water will be containerized for subsequent analysis and disposal, as described in section 7.0 of the WP..

3.7.3 Monitoring Well Purging

The objective is to purge monitoring wells until turbidity stabilizes to a level as low as possible, and this parameter will be given the greatest weight in determining when groundwater sampling may begin. A low-flow pump will be utilized to avoid intrusion of particulates within the well or from the surrounding formation. Groundwater from each well will be purged until parameters have stabilized. A turbidity level of 5 NTUs or less is the well purging goal, but not an absolute value before sampling. Other field parameters including temperature, conductivity, pH, and DO will also be monitored. As practical, all field measurements will be taken from the flow cell and will be recorded during and after purging, and before sampling. Field parameters should generally be within ±10 percent for three consecutive readings, at a minimum of one minute apart, so that it may be determined when the parameters stabilize.

Upon opening each monitoring well and point, the concentration of ionizable vapors in the headspace will be measured using a PID and water level measurements will be recorded using an electronic oil-water interface probe. The depth to product (if present), depth to water, and the total depth will be measured from the top of the marked PVC casings. The volume of water in the well will be calculated so that the number of well volumes purged and an estimate of the time required to purge the well can be made. Before sampling, the wells will be purged utilizing a low-flow submersible stainless steel pump using dedicated Teflon® or Teflon®-lined polyethylene tubing connected to a flow cell. Very low purging rates are proposed, on the order of 100 ml/minute to 500 ml/minute, to minimize suspension of particulate matter in the well.

Purging will be done with the pump placed near the top of the water column to insure that all stagnant water in the well is removed, while not stirring sediment that may have accumulated on the bottom of the well. Equipment will be lowered into the well very carefully to prevent suspension of bottom sediment and subsequent entrainment onto sampling equipment. Surging will be avoided. Pumps must be carefully cleaned between wells according to the procedures specified in Section 2.1.2.3 of the QAPP. It is anticipated that no more than three well volumes will be purged in order for turbidity to reach a minimum and the other parameters to stabilize. Ideally, pumping rates will be at a rate so that no draw-down of the groundwater level occurs (i.e. pumping rate equals recharge rate). During purging, ERT will actively monitor and track the volume of water purged and the field parameter readings. Data will be recorded in the field logbook and the groundwater purge datasheet (Attachment 1).

In the event that the entrained purge water becomes frozen during the purging process, and traditional low-flow methodology is impractical, on-site dedicated bailers will be utilized to complete the purging process. If this method is required to purge due to weather conditions, a minimum of three well volumes will be purged or until the well is purged dry. The total volume

purged by hand bailing will be based on the standing well volume calculated from depth to water, total depth measurement, and a well diameter multiplication variable. If well recharge is insufficient to purge three volumes the well will be allowed to recharge a reasonable amount of time prior to sampling. Well purging water will be containerized for subsequent analysis and disposal.

3.7.4 Groundwater Monitoring Well Sampling

Once groundwater conditions have stabilized and groundwater levels have recovered, all samples will be collected from the flow cell outlet (connected to the low-flow submersible pump). All sampling equipment will be cleaned according to the procedures specified in Section 4.11 of the QAPP.

Sampling will be performed by suspending the bottom of the pump and/or bailer a minimum of 15 inches above the bottom of the well and a minimum of 6 inches from the top of the water level, in order to minimize the amount of suspended sediment in the sample. Pumping rates for withdrawing the samples will be similar to those followed for well purging.

The samples will be collected in sample bottles (pre-preserved, if appropriate), placed in iced coolers and removed from light immediately after collection. When collecting samples, the water should be allowed to run down the side of the collection bottle. In addition, all samples bottles must be filled to the top so that no aeration of the samples occurs during transport. All bottles will be filled to avoid cascading and aeration of the samples, the goal being to minimize any precipitation of colloidal matter. No head space or air bubbles will be left in the sample containers. Samples for dissolved metals will be collected in unpreserved containers and will be filtered and preserved at the laboratory within 24 hours of sampling. Sample filtration for metals will be performed at the lab.

Groundwater samples will be analyzed for TOC (Lloyd Khan method), Total Organic Halides (TOX) (Method 9020), Nitrate/Nitrite (Method 353.2), total and dissolved TAL Metals (Methods 6010B/7470), Total Cyanide (Method 9010A or 9012+C23), Explosives (Method 8330), Nitroguanidine (Method 8330m), Nitroglycerine (Method 8330m), Nitrocellulose (Method UV/VIS), VOCs (Method 8260B), TCL Pesticides (Method 8081A), PCBs (Method 8082), and Perchlorate (Method 6850).

3.8 QA/QC Sampling

QA/QC sampling is essential to the collection of justifiable RI data. QA/QC samples to be collected for laboratory analyzed samples include duplicate samples, field blanks (FB), equipment rinsate blanks (ERB), trip blanks (TB) and matrix spikes (MSs)/matrix spike duplicates (MSDs). One trip blank will be sent for laboratory analysis within each sample cooler. Duplicate samples, FBs, ERBs and MSs/MSDs will be collected and delivered for laboratory analysis at a minimum frequency of 1 per 20 RI samples. A more detailed description of the QA/QC samples and methods is provided in Section 2.4 of the QAPP.

QA/QC procedures for field screened samples incorporate the use of duplicate sampling at a rate of at least 1 per 20 field screened samples. In addition to the field QC, internal QA checks are

performed by the laboratory to verify the quality of the measurements of laboratory investigations and associated tasks. Types of laboratory QA checks are specified in the QAPP.

3.9 Waste Characterization Sampling

Sampling activities will be performed in such a manner so as to minimize the generation of IDW. Those wastes generated will be handled utilizing a prudent and reasonable approach to decisions related to the management of the IDW.

Several types of IDW are expected to be generated during the field activities associated with the RI, including:

- Rinsate from equipment decontamination procedures
- Purge water from newly installed groundwater monitoring well (MW-11)
- Ordnance-related scrap recovered during the surface clearance
    - non-hazardous metal scrap
    - metal scrap with explosive residue
- Soil cuttings from the installation of MW-11
- Disposable PPE

The IDW that will require sampling is discussed below. Additional details on IDW management are presented in section 7.0 of the WP.

3.9.1 Metal (Solid) Waste

Metal scrap, collected during the MEC clearance activities and test pit excavations will be field screened immediately after collection. Scrap with no MEC constituents (as determined by the field screening methodology), will be placed in a 55-gallon drum (labeled "clean"). Scrap that test positive for MEC constituents will be placed into a separate 55-gallon drum (labeled "MEC"). The field screening of metal scrap will be performed as discrete (grab) samples.

3.9.2 Soil (Solid) Waste

Soil cuttings recovered during the installation of MW-11 will be containerized in a 55-gallon drum. Waste soil will be sampled by advancing a hand auger through the containerized volume of soil (not just grabbing a sample off the top) and characterized for TCLP analyses and RCRA characteristics. If other analyses become necessary, the parameters and methods will be provided as an attachment to the QAPP.

3.9.3 Liquid Waste

Liquid waste sampling methods to be employed during the RI include either dedicated dippers or glass tube samplers for sampling of drummed equipment decontamination water or monitoring well development/purge water. Dippers are used to collect samples from the surface of the liquid, and are appropriate for wastes that are homogeneous. Glass tube samplers consist of glass tubes of varying length and diameter used to collect a full-depth liquid sample from a drum or similar container. Liquid wastes will be sampled as grab samples (when there is only one drum of a particular liquid waste) or as a composite sample, when there is more than one drum of the same type of waste (e.g., decon water, purge water or development water).

3.10 Decontamination of Field Equipment

The purpose of developing and adhering to an adequate decontamination process is to prevent or reduce cross-contamination between samples and media. Section 2.1 of the QAPP describes in detail the equipment and procedures that have been developed to prevent cross contamination of potential contaminants to samples, and protect the health and safety of site personnel.

3.11 Sample Handling

Proper sample management includes procedures which dictate sample handling to ensure analysis validity. Certain procedures are considered vital to the qualification of data generated from the collection and analysis of environmental samples. Procedures detailed in the QAPP include proper sample custody, establishment of a chain-of-custody, adherences to a standard sample naming scheme, application of a custody seal and the transfer of custody, and properly packaging and shipment of samples to ensure uncompromised delivery to the laboratory.

3.12 Documentation of Field Activities

It is essential to detail all field activities associated with the RI. Documentation will be the responsibility of each member of the field team. A standard operating procedure for correct field documentation techniques is provided in the QAPP. Bound, hard cover books will be used daily to record typical events during the RI. Other documents may be utilized to perform the logging of samples, bore holes, well installation, groundwater sampling and other such activities. If necessary, field memorandums may be typed and distributed to field personnel, USACE Project Manager and other pertinent recipients, and will be considered official documentation of the particular event described.

3.13 Sample Documentation/Chain-of-Custody Procedures

3.13.1 Field Logbook

All information related to sampling activities will be recorded in the field logbook. The logbook will also contain any deviations to the FSP that arise based on field conditions. At a minimum, the information provided in the SOP detailed in the QAPP will be addressed in the logbook.

All entries in the field logbook will be made legibly with indelible ink. Entries will be objective, factual, and free of personal feelings or other terminology that might prove inappropriate. No blank pages or sections of pages will be included in the logbook. If a page is not completely filled in, a line will be drawn through the blank portion and initialed by the person keeping the log. Errors in the logbook will be corrected by drawing a single line through the error, and initialing and dating the correction. The field logbook will serve as a permanent record of the field activities and will become part of the project files.

3.13.2 Sample Labeling

All samples will be properly labeled with the label affixed to the container before transportation to the laboratory. The label will be filled out with permanent ink or marker, and printed legibly (see Attachment 1). Labels will be covered with clear packing tape to ensure the integrity of the transcribed information. A SOP for proper sample labeling is included in the QAPP.

3.13.3 Sample Identification

A sample numbering system will be used to identify each sample collected and submitted for analysis to assist in the tracking of samples and to facilitate retrieval of analytical results. The sample identification numbers for each sampling effort will be used on sample labels or tags, chain-of-custody (COC) forms, field logbooks, field sheets and all other applicable documentation. A listing of all sample identification numbers will be recorded in the field logbook.

3.13.4 Chain-of-Custody Records

COC procedures document the handling of each sample from the time it is collected through transportation, sample receipt by the laboratory, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in the field and laboratory. COC procedures will be implemented so that a record of sample collection, transfer of samples between personnel, sample shipping, and receipt by the laboratory is maintained. The COC record serves as a legal record of possession of the sample. A detailed description of the SOP for COC is provided in the QAPP and an example COC form is provided in Attachment 1.

The COC record will be initiated with the acquisition of the first sample. The COC record will remain with the sample at all times and will bear the name of the person assuming responsibility for the samples. The Site Field Manager will be responsible for ensuring secure and appropriate handling of the bottles and samples.

The COC record will be signed by the sampler and then signed again by the receiving individual at the laboratory. Both the sampler and the laboratory will retain a copy of the COC record.

In addition to the COC record, custody seals will be used. A custody seal is an adhesive seal placed on the sample cooler in such a way that it must be broken to open the cooler. The custody seal ensures that no tampering of the samples occurred between the field and receipt by the laboratory.

3.13.5 Laboratory Sample Custody and Documentation

Laboratory sample custody and documentation SOP is detailed in the QAPP and CT Laboratory Quality Assurance Manual (QAM).

3.13.6 Sample Packaging and Shipping

It is anticipated that all samples collected will be considered environmental samples and will be handled as such. Environmental samples are defined as those samples collected from environmental matrices such as soil, groundwater, or sediments that are not considered high-hazard samples. Environmental samples will be packaged for shipment in general accordance with Appendix F of EM 200-1-3 and as detailed in the QAPP.

3.14 Sampling Apparatus and Field Instruments

Field instruments will be calibrated, operated, and maintained in accordance with manufacturer's instructions and specific instrument SOPs. Daily, and in some cases more frequently, calibration

of equipment will provide QC checks on all equipment used during the performance of project activities. Each instrument will have an individual identification number affixed. This number will be transcribed on field data records when using a particular instrument for a sampling event. All calibration, repair, and service records will be kept in individual equipment logbooks maintained for each instrument. Equipment that consistently falls out of calibration or exceeds manufacturer's critical limits will be repaired or replaced.

3.14.1 Photo Ionization Detector

A PID uses photo ionization to detect compounds that have an ionization potential below the energy of the lamp installed in the instrument. Lamp energy is measured in electron volts (eV) and the PID that will be utilized will be equipped with an 11.2-eV lamp. The PID instrument requires calibration before use, using commercially available gases (isobutylene calibration gas [or equivalent]) of known concentrations. Calibration will be performed daily according to the manufacturer's recommendations and will be recorded in the appropriate equipment logbook or calibration form.

3.14.2 Equipment Decontamination

It is anticipated that only a water level meter and water quality indicator will require decontamination, as dedicated tubing will be used with a peristaltic pump, or a dedicated stainless steel bailer will be used to collect samples. Measuring equipment will be decontaminated utilizing the SOP detailed in the QAPP.

Any groundwater sampling equipment that is not dedicated or disposable sampling apparatus will be decontaminated prior to and after use, utilizing the SOP detailed in the QAPP.

3.14.3 Equipment Maintenance and Calibration

Calibration procedures for field instrumentation are performed to ensure that the instruments are operating properly and produce data that can satisfy the objectives of the sampling program. This screening level data is used to monitor worker health and safety and to detect organic vapors within the monitoring wells. A PID will be used during all sampling activities.

To ensure that the instruments are operating properly and are producing accurate and reliable data, routine calibration must be performed before and during use. ERT will ensure that factory calibrations are performed at the frequency recommended by the manufacturer. Field calibrations will be performed at the beginning of each day. Calibrations will be checked if grossly inaccurate readings are evident. If necessary, the instrument will be returned to the vendor for immediate repair and servicing and a backup instrument will be procured in an expeditious manner.

PIDs can measure total organic vapors and are highly sensitive to aromatic compounds, moderately sensitive to unsaturated chlorinated compounds, and less sensitive to aliphatic hydrocarbons. The instrument can respond to organic compounds with ionization potentials less than the rated eV of the ultraviolet bulb in the unit. Due to its longevity and range of detectable contaminants, the most frequently used ultraviolet bulb is a 10.2-eV. However, in order to detect the whole suite of VOC's, an 11.2-eV bulb will be used.

Several manufacturers produce PIDs for field monitoring of airborne VOCs. The field technician will follow the manufacturer's calibration requirements. General guidance for PID calibration includes:

- Factory service and calibration once per year; and
- For any PID instrument, a two-point calibration before daily use (UHP air and a representative concentration of isobutylene in air standard).

3.15 Nonconformance/Corrective Actions

Deficiency tracking and corrective actions will be taken whenever there is a deficiency in characteristic(s), documentation, or procedures that result in the quality of an item being deemed unacceptable or indeterminate with respect to specified criteria. Nonconformance examples include test failure, physical defects, data losses, deviations from prescribed project plans, and deviations from industry-accepted processes and procedures. If nonconformance is related to a hazardous condition or potential safety concerns, corrective action will immediately be taken and documentation will be completed as an immediate follow-up. Prompt corrective action for safety issues will never be impeded by the need to process a deficiency report.

3.15.1 Response

Corrective action will be initiated when potential or existing conditions are identified that may adversely impact data quality or quantity. It is the responsibility of the individual who first recognizes an out-of-control event to initiate corrective action and to document the action. Notification of the nonconformance will be directed to the individual's supervisor. It is the supervisor's responsibility to coordinate corrective action with the Project Manager and to follow up to ensure re-establishment of control. Events that require corrective action include:

- Violation of established field or sample handling procedures
- Violation of established analytical controls
- Violation of established field or sample collection procedures
- Violation of established sample shipping or receiving procedures
- Results of performance, system, or project QA audits
- Data reporting errors.

When a deficiency is detected, a deficiency report will be generated, describing and documenting the occurrence and the corrective action taken or suggested to be taken. The appropriate criteria will be cited and sufficient supporting data will be provided to allow a resolution to the deficiency. The report will be generated by the Site Field Manager, and will be provided to the Project Manager for review and approval of the proposed corrective action. The report will describe the cause for the deficiency, corrective action taken, measures taken to prevent a recurrence, and the date actions are to be completed. The PM will review and sign the deficiency report.

The deficiency will be reported to the Contracting Officer's Representative (COR) within 48 hours. Correction of the deficiency will be verified and the deficiency report indicating closeout will be signed by the Site Manager. The deficiency report will not be closed out until all corrective and preventive measures have been completed.

The Site Manager will maintain a deficiency log of all open deficiencies, date of issue, responsible organization or individual, date of anticipated corrective action, and closed-out deficiency reports. The log will provide the deficiency number, a brief description of the action, and the date closed. Records to be retained in the project file will contain pertinent information necessary to document resolution of the deficiency including scope and significance of the problem.

Corrective action may take several forms, but the following steps are almost always included:

- Check the calculations
- Check the instrument for proper setup
- Re-analyze the control item
- Stop work (if necessary).

The corrective action may be immediate or long term. A corrective action requiring immediate response may be recalibration, recalculation, reanalysis, or repeating sample collection. Long-term corrective action may be identified through, but not limited to, performance evaluation samples, standards, and control charts. Corrective actions will be documented on a Field Change Report and maintained in project files.

The Field Change Report includes a description of the corrective action planned and has space for follow-up. The Project Manager will verify that initial action has been taken and appears to be effective and, at an appropriate later date, will check to see if the problem has been resolved. The Project Manager receives a copy of all Field Change Reports and enters them into the Corrective Action Log. This permanent record will aid the Project Manager in following up and will assist in resolving QA problems. An example Field Change Request form is included in Attachment 1.

3.15.2 Re-establishment of Control

Immediate corrective action is usually applied to real-time, nonrecurring problems. Instrument and equipment malfunctions and nonconforming field procedures are examples of problems amenable to this type of action. The individual who observes nonconformance to previously established criteria or protocols involving equipment, instruments, data collection or handling, or field methods will immediately notify the Site Manager and/or the Project Manager, who will investigate the extent of the problem and take the necessary corrective steps. The Site Manager will prepare a corrective action report for the Project Manager. These individuals will collectively decide how to proceed.

Once the immediate corrective action is implemented, the task leader will monitor the situation closely for recurrence to ensure that the corrective action has been effective and the problem is

no longer occurring. If the problem continues, the corrective action may be deemed ineffective, and additional or alternative corrective actions may be necessary. In such cases, continued consultation with the Site Manager and the Project Manager will occur, as necessary, to collectively monitor and resolve the problem in a timely manner.

Long-term corrective action procedures are devised and implemented to prevent the recurrence of a potentially serious problem. The Site Manager will be notified of the problem and will investigate the severity and extent of the problem. A corrective action report will be filed with the Project Manager.

3.15.3 Documentation

The Site Manager or the Project Manager documents all notifications, recommendations, and the agreed-upon corrective action plan, if required. The Project Manager notifies project staff and implements the agreed-upon course of action. The Site Manager verifies the efficiency of the implemented actions. The development and implementation of preventive and corrective actions will be timed, to the greatest extent possible, to not adversely impact either project schedules or subsequent data-generation/processing activities. The Site Manager will also be responsible for developing and implementing routine program controls to minimize the need for corrective action.

3.16 Field Assessment-Three Phase Inspection Procedures

ERT will ensure that quality is maintained by implementing a three-phase inspection process as discussed in engineering regulation ER 1180-1-6, "*Construction Quality Management*", dated September 1995. The three QC phases used during this process are preparatory, initial, and follow-up.

3.16.1 Preparatory

The preparatory phase will include review of scope of work, planning documents, work requirements, and field sampling DQOs by the project team. The Project Manager will review work requirements; perform a physical examination of required materials, equipment, and work areas; and, if necessary, will perform a demonstration of equipment to be used and forms to be completed, including the COC, and other field notes.

3.16.2 Initial Phase

The initial phase will include 100% review of field tasks during initial sampling effort by the Project Manager and field personnel to insure tasks are being completed in compliance with the planning documents such that DQOs will be obtained. This phase will be performed when sampling is first initiated. Initial instrument calibrations will be observed.

3.16.3 Follow-Up Phase

Follow-up phase inspections will be accomplished on an as-needed basis to ensure compliance with contract requirements. General procedures and documentation will be inspected to ensure continued compliance with contract requirements.

The follow-up phase will consist of review and resolution of problems or issues occurring during sampling and analysis that were not anticipated or included in the planning documents. Follow-up phase activities will be performed by the Project Manager. Follow-up will also include intermittent quality review of field practices, as well as initiation of possible non-conformance memoranda, if necessary. Non-conformance memoranda will also be initiated if problems are discovered during USACE oversight of field activities.

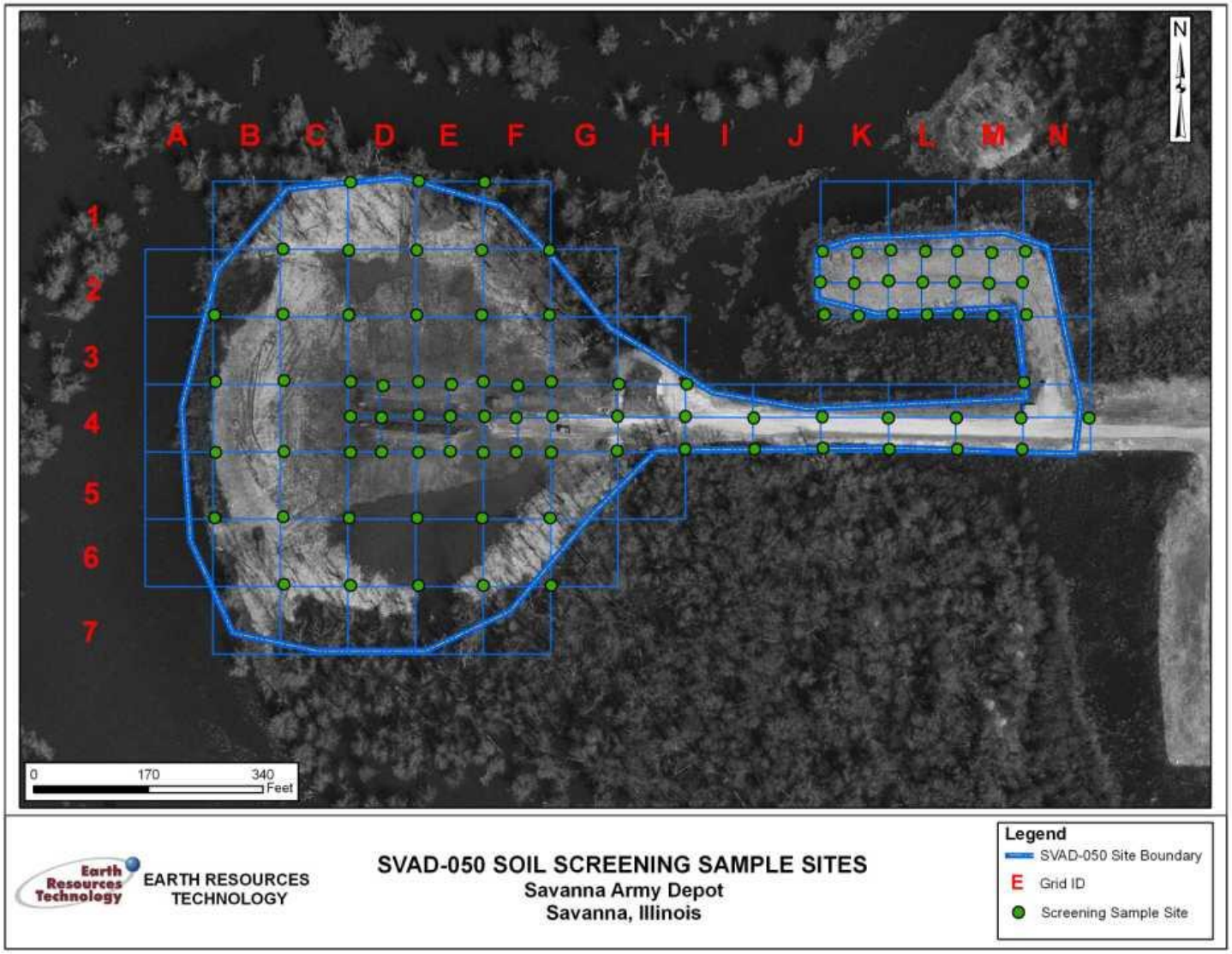

Figure 3-1. Proposed Grid System for Phase I Systematic Soil Screening Sampling

Figure 3-2. Proposed Location for New Groundwater Monitoring Well

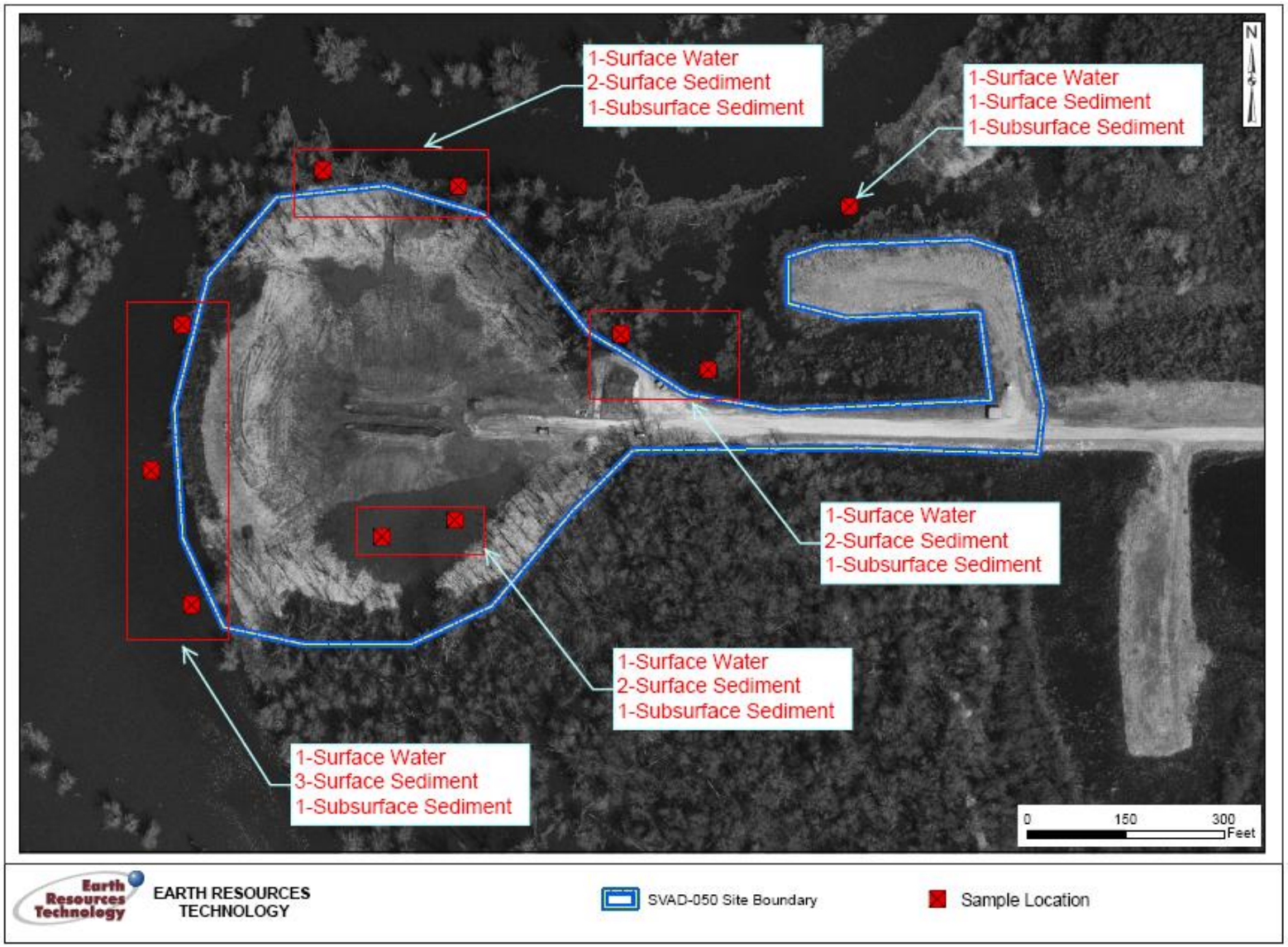

Figure 3-3. Proposed Locations for Sediment and Surface Water Sampling

Table 3.1. Summary of Proposed RI Samples

Sample Type	Matrix	Phase of Sampling	Total Number of Samples	Surface Samples	Subsurface Samples	Duplicate Samples	ERB Samples*	FB Samples*	MS/MSD Samples*
DropEx Soil Screening	Soil	Phase I	116	105	0	11	0	0	0
Test Pit (Soil)	Soil	Phase I	15	6	6	1	1	0	1
Biased	Soil	Phase II	59	25	25	5	2	0	2
Sediment	Sediment	Phase II	19	10	5	2	1	0	1
Groundwater[1]	Aqueous	Phase II	3	N/A	1	1	0	0	1
Surface Water	Aqueous	Phase II	9	5	N/A	1	1	1	1

ERB Equipment Rinsate Blank
FB Field Blank
MS/MSD Matrix spike/Matrix spike duplicate
[1] All groundwater and associated QC samples from MW-11 will be sampled in conjunction with the fourth quarter 2009 groundwater sampling event at the OB/OD area

Table 3.2. Analytical Parameters, Methods, Preservation and Container Requirements

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Soil/Sediment	VOCs (TCL)	Grab	77	SW-846 Method 8260B	Cool to 4^0 C; no headspace	14 days to analysis	(1) Terra Core kit (Low-level)
Soil/Sediment	PCBs	Grab	77	SW-846 Method 8082	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(1) 9-oz amber glass jar
Soil/Sediment	Pesticides (TCL)	Grab	77	SW-846 Method 8081A	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	SVOCs (TCL)	Grab	77	SW-846 Method 8270C	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediments	Metals (TAL)	Grab	77	SW-846 Method 6010B/7470A	Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 9-oz glass jar
Soil/Sediments	Cyanide	Grab	77	SW-846 Method 9012A	Cool to 4^0 C	14 days to analysis	
Soil/Sediments	Explosives	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(2) 9-oz glass jars
Soil Sediments	Nitrocellulose	Grab	77	SW-846 Method 9056M	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroguanidine	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroglycerine	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	7 14 days to extraction; 40 days from extraction to analysis	

Table 3.2. Analytical Parameters, Methods, Preservation and Container Requirements

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Groundwater/Surface Water	VOCs (TCL)	Grab	6	SW-846 Method 8260B	pH<2 with HCl; Cool to 4^0 C; no headspace	14 days to analysis	(5) 40 mL VOA vials
Groundwater/Surface Water	SVOCs (TCL)	Grab	6	SW-846 Method 8270C	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater/Surface Water	Metals- total (TAL)	Grab	6	SW-846 Method 6010B	pH<2 with HNO_3; Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Groundwater/Surface Water	Metals-dissolved (TAL)	Grab	6	SW-846 Method 6010B/7470A	pH<2 with HNO_3; Cool to 4^0 C	24 hours to filtering and preservation (pH<2 with HNO_3); 28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Groundwater/Surface Water	Total Cyanide	Grab	6	SW-846 Method 9012A	pH>12 with NaOH; Cool to 4ºC	14 days to analysis	(1) 500 mL polyethylene container
Groundwater/Surface Water	Nitrate+Nitrite	Grab	6	SW-846 Method 9056	pH<2 with H_2SO_4; Cool to 4^0 C	28 days to analysis	(1) 125 mL polyethylene bottle
Groundwater/Surface Water	Pesticides (TCL)	Grab	6	SW-846 Method 8081A	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater/Surface Water	PCBs	Grab	6	SW-846 Method 8082	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(1) 1L amber glass jar
Groundwater/Surface Water	Explosives	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	(2) 1L amber glass bottle
Groundwater/Surface Water	Nitroguanidine	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	

Table 3.2. Analytical Parameters, Methods, Preservation and Container Requirements							
Sample Matrix	**Analytical Parameter**	**Sample Type[1]**	**No. of Samples[2]**	**EPA Analytical Method**	**Sample Preservation**	**Holding Time[3]**	**Sample Container[4,5]**
Groundwater/Surface Water	Nitroglycerine	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Groundwater/Surface Water	Nitrocellulose	Grab	6	SW-846 Method 9056M	Cool to 4ºC	7 days to analysis	
Groundwater	Perchlorate	Grab	1	SW-846 Method 6850	Cool to 4ºC	28 days to analysis	(1) 125 mL polyethylene container
Investigative Derived Waste (IDW) – Soil/Solids	TCLP VOC (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/8260B	Cool to 4^0 C	14 days to TCLP extraction; 14 days from TCLP extraction to analysis	(1) 32-oz glass jar (1) 9-oz glass jar
Investigative Derived Waste (IDW) - Soil/Solids	TCLP SVOC (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/ 8270C	Cool to 4^0 C	14 days to TCLP extraction; 7 days from TCLP extraction to SVOC extraction; 40 days from SVOC extraction to analysis	
Investigative Derived Waste (IDW) - Soil/Solids	TCLP Pesticides (RCRA)	Grab	TBD/ up to 4[6]	SW-846 Methods 1311/8081A	Cool to 4ºC	14 days to TCLP extraction; 7 days from TCLP extraction to pesticide extraction; 40 days from pesticide extraction to analysis	
Investigative Derived Waste (IDW) - Soil/Solids	TCLP Metals (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/ 6010B/7470A	Cool to 4^0 C	Hg: 28 days to TCLP extraction; 28 days from TCLP extraction to analysis Other Metals: 6 months to TCLP extraction; 6 months from TCLP extraction to analysis	
Investigative Derived Waste (IDW) - Soil/Solids	Ignitability	Grab	TBD/ up to 4[6]	SW-846 Method 1010/1030	Cool to 4^0 C	None specified	

Table 3.2. Analytical Parameters, Methods, Preservation and Container Requirements

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Investigative Derived Waste (IDW) - Soil/Solids	Corrosivity	Grab	TBD/ up to 4[6]	SW-846 Method 9045C	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) - Soil/Solids	Reactive cyanide	Grab	TBD/ up to 4[6]	SW-846 Chapter 7, Section 7.3.3	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) - Soil/Solids	Reactive sulfide	Grab	TBD/ up to 4[6]	SW-846 Chapter 7, Section 7.3.4	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP VOC (RCRA)	Grab	TBD	SW 846 Methods 1311/8260B	Cool to 4^0 C	14 days to TCLP extraction; 14 days from TCLP extraction to analysis	(3) 1L glass amber bottles (2) 500mL polyethylene bottle (1) 250mL polyethylene bottle
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP SVOC (RCRA)	Grab	TBD	SW 846 Methods 1311/ 8270C	Cool to 4^0 C	14 days to TCLP extraction; 7 days from TCLP extraction to SVOC extraction; 40 days from SVOC extraction to analysis	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP Pesticides (RCRA)	Grab	TBD	SW-846 Methods 1311/8081A	Cool to 4ºC	14 days to TCLP extraction; 7 days from TCLP extraction to pesticide extraction; 40 days from pesticide extraction to analysis	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP Metals (RCRA)	Grab	TBD	SW 846 Methods 1311/ 6010B/7470A	Cool to 4^0 C	Hg: 28 days to TCLP extraction; 28 days from TCLP extraction to analysis Other Metals: 6 months to TCLP extraction; 6 months from TCLP extraction to analysis	
Investigative Derived Waste (IDW) – Decontamination Liquid	Ignitability	Grab	TBD	SW-846 Method 1010/1030	Cool to 4^0 C	None specified	

Table 3.2. Analytical Parameters, Methods, Preservation and Container Requirements

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Investigative Derived Waste (IDW) – Decontamination Liquid	Corrosivity	Grab	TBD	SW-846 Method 9045C	Cool to 4^0 C	As soon as possible (within 3 days of collection)	
Investigative Derived Waste (IDW) – Decontamination Liquid	Reactive cyanide	Grab	TBD	SW-846 Chapter 7, Section 7.3.3	Cool to 4^0 C	As soon as possible (within 3 days of collection)	
Investigative Derived Waste (IDW) – Decontamination Liquid	Reactive sulfide	Grab	TBD	SW-846 Chapter 7, Section 7.3.4	Cool to 4^0 C	As soon as possible (within 3 days of collection)	

[1] For soil samples, a six-inch sampling interval is the preferred sample size; however, sample volume recovery, analytical method requirements, and field conditions can affect the actual sample interval size. For these reasons, the actual sampling interval may change in order to obtain adequate volume.
[2] Actual number of samples may vary depending on field conditions, sample material availability, and field observations. **Does not include QA samples**.
[3] From date of sample collection
[4] I-Chem Series 300 bottles
[5] MS/MSDs require duplicate volume for all parameters for solid matrices; MS/MSDs require triplicate volume for organic parameters for aqueous matrices and duplicate volume for inorganic parameters for aqueous matrices
[6] Up to 4 soil samples collected during Phase II may be analyzed using TCLP.
TBD = To Be Determined

Table 3.3. QC Sample Preservation and Container Requirements

Sample Matrix	Analytical Parameter	QC Type	No. of Samples	EPA Analytical Method	Sample Preservation	Holding Time[1]	Sample Container[2] per QC Type
Soil/Sediment	VOCs (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8260B	Cool to 4^0 C; no headspace	14 days to analysis	(1) Terra Core kit (Low-level)
Soil/Sediment	PCBs	Field Duplicate	1 per 20 Samples	SW-846 Method 8082	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(1) 9-oz amber glass jar
Soil/Sediment	Pesticides (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8081A	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	SVOCs (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8270C	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Metals (TAL)	Field Duplicate	1 per 20 Samples	SW-846 Method 6010B/7000 Series	Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 9-oz glass jars
Soil/Sediment	Cyanide	Field Duplicate	1 per 20 Samples	SW-846 Method 9012A	Cool to 4^0 C	14 days to analysis	
Soil/Sediment	Explosives	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(2) 9-oz glass jars
Soil/Sediment	Nitrocellulose	Field Duplicate	1 per 20 Samples	SW-846 Method 9056M	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroguanidine	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroglycerine	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	

Table 3.3. QC Sample Preservation and Container Requirements

Sample Matrix	Analytical Parameter	QC Type	No. of Samples	EPA Analytical Method	Sample Preservation	Holding Time[1]	Sample Container[2] per QC Type
Surface Water	VOCs (TCL)	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8260B	pH<2 with HCl; Cool to 4^0 C; no headspace	14 days to analysis	(5) 40 mL VOA vials
Surface Water	SVOCs (TCL)	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8270C	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Surface Water	Metals- total (TAL)	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 6010B/7000 Series	pH<2 with HNO_3; Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Surface Water	Metals-dissolved (TAL)	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 6010B/7000 Series	Cool to 4^0 C	24 hours to filtering and preservation (pH<2 with HNO_3); 28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Surface Water	Cyanide	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	EPA Method 335.3	pH>12 with NaOH; Cool to 4ºC	14 days to analysis	(1) 500 mL polyethylene container
Surface Water	Nitrate+ Nitrite	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	EPA Method 353.2/SM 4500-NO_2B (18th edition)	pH<2 with H_2SO_4; Cool to 4^0 C	28 days to analysis	(1) 125 mL polyethylene bottle
Surface Water	Pesticides (TCL)	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8081A	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle

Table 3.3. QC Sample Preservation and Container Requirements

Sample Matrix	Analytical Parameter	QC Type	No. of Samples	EPA Analytical Method	Sample Preservation	Holding Time[1]	Sample Container[2] per QC Type
Surface Water	PCBs	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8082	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(1) 1L amber glass jar
Surface Water	Explosives	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	(2) 1L amber glass bottle
Surface Water	Nitroguanidine	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	(2) 1L amber glass bottle
Surface Water	Nitroglycerine	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Surface Water	Nitrocellulose	Field Duplicate Equip Blank Rinse Blank	1 per 20 Samples per QC Type	SW-846 Method 9056M	Cool to 4ºC	7 days to analysis	
Groundwater	VOCs (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8260B	pH<2 with HCl; Cool to 4^0 C; no headspace	14 days to analysis	(5) 40 mL VOA vials
Groundwater	SVOCs (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8270C	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater	Metals- total (TAL)	Field Duplicate	1 per 20 Samples	SW-846 Method 6010B/7000 Series	pH<2 with HNO_3; Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle

Table 3.3. QC Sample Preservation and Container Requirements

Sample Matrix	Analytical Parameter	QC Type	No. of Samples	EPA Analytical Method	Sample Preservation	Holding Time[1]	Sample Container[2] per QC Type
Groundwater	Metals-dissolved (TAL)	Field Duplicate	1 per 20 Samples	SW-846 Method 6010B/7000 Series	Cool to 4^0 C	24 hours to filtering and preservation (pH<2 with HNO_3); 28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Groundwater	Cyanide	Field Duplicate	1 per 20 Samples	EPA Method 335.3	pH>12 with NaOH; Cool to 4ºC	14 days to analysis	(1) 500 mL polyethylene container
Groundwater	Nitrate+ Nitrite	Field Duplicate	1 per 20 Samples	EPA Method 353.2/SM 4500-NO_2B (18th edition)	pH<2 with H_2SO_4; Cool to 4^0 C	28 days to analysis	(1) 125 mL polyethylene bottle
Groundwater	Pesticides (TCL)	Field Duplicate	1 per 20 Samples	SW-846 Method 8081A	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater	PCBs	Field Duplicate	1 per 20 Samples	SW-846 Method 8082	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(1) 1L amber glass jar
Groundwater	Explosives	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	(2) 1L amber glass bottle
Groundwater	Nitroguanidine	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Groundwater	Nitroglycerine	Field Duplicate	1 per 20 Samples	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Groundwater	Nitrocellulose	Field Duplicate	1 per 20 Samples	SW-846 Method 9056M	Cool to 4ºC	7 days to analysis	
Groundwater	Perchlorate	Field Duplicate	1 per 20 Samples	SW-846 Method 6850	Cool to 4ºC	28 days to analysis	(1) 125 mL polyethylene container

[1] From date of sample collection
[2] I-Chem Series 300 bottles

4.0 REFERENCES

U.S. Army Corps of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3. February 1. http://www.usace.army.mil/inet/usace-docs/eng-manuals/em200-1-3/toc.htm

ATTACHMENT 1
Forms

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FCR# _____
Date_____
FIELD CHANGE REPORT

1. What is needed?

2. Why is it required? What impact if not done?

3. How much does it cost (unit, material, time, etc.)?

4. What is the bottom-line cost (overhead, indirect, fee, etc.)?

5. Where do we stand in relation to original proposal (what will be affected and by how much)?

_____ _____
Site Manager Date

_____ _____
Project Manager Date

Monitoring Well: _____

Groundwater Sampling Log

Sheet ____ of ____

Site: _____ Consulting Firm: _____
Date: _____ Field Personnel: _____
Weather: _____ _____

Well Permit #: _____ Well Depth _____ Screened/Opened _____
Well Diameter _____ (inches)

PID/FID Readings Background: _____ Pump Intake Depth: _____ ft below TOC
Beneath Outer: _____ Depth to Water Before Pump Installation: _____ ft below TOC
Beneath Inner: _____

Time	Purging	Sampling	pH (pH units)		Conductivity (ms/cm)		Turbidity (NTU)		DO (mg/L)		Temperature (degrees C°)		ORP (mv)		Pumping Rate (mL/min)	Depth to Water (ft below TOC)
			Reading	Change	Reading	%Change	Reading	%Change	Reading	Change	Reading	Change	Reading	Change		
				NA		NA		NA		NA		NA		NA		

Total Volume Purged:

Sample ID: Jarware Collected:

Sample Time: Sample Analysis:

Stabilization Criteria
D.O. +/- .3 mg/l
Turb. +/- 10 %
Cond. +/- 3 %
ORP +/- 10 Mv
pH +/-0.1 unit

2 inch wells = 0.163 g/ft
4 inch wells = 0.653 g/ft

ERT, Inc
10810 Guilford Road Suite 105
Annapolis Junction, MD

CHAIN OF CUSTODY

GPL Project Manager:	Client Name:	Phone:	
Project Name:	Address:	Cell:	
Purchase Order:	City, State	Zip Code	

Comments: H=Hold Analysis Request X=Analyze

Preservatives and Containers

Sample Information | **Methods for Analysis** | **RUSH**

No.	Client Name, address and phone #	Date Sampled	Time Sampled	Matrix	Sampler's Initials															QC (MS/MSD)	TOTAL BOTTLES
1							X	X	X	X	X	X	X	X	X	X	X						4
2																-							
3																							
4																							
5																							
6																							
7																							
8																							
9																							
10																							
11																							
12																							

Sample Matrix: WG= Groundwater; SO= Soil; WS= Surface Water; AA= Ambient Air; WQ= Water Quality Total number of samples

CUSTODY	For Lab Use	COOLER RECEIPT CONDITION
Time Date Received By:	**WORK ORDER #:_____**	
Time: Date: Relinquished By:		
Time: Date Received By:		
Time: Date: Relinquished By:		
Time: Date Received By:		

Example Custody Seal

GPL Laboratories

CLIENT/SOURCE	☐ GRAB ☐ COMPOSITE ☐ OTHER:
SITE NAME	DATE
SAMPLE #	TIME
ANALYSIS	PRESERVATIVE
	COLL BY

Example Sample Label

APPENDIX E
QUALITY ASSURANCE PROJECT PLAN

FINAL
QUALITY ASSURANCE PROJECT PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT
OPEN BURN/OPEN DETONATION AREA (SVAD-050)
SAVANNA ARMY DEPOT ACTIVITY
SAVANNA, ILLINOIS

Prepared for

U.S Army Corps of Engineers
Louisville District

Contract Number: W912QR-08-D-0012
Delivery Order 0001

Prepared by

Earth Resources Technology, Inc.
4890 University Square, Suite 2A
Huntsville, Alabama 35816
(256) 217-8991

Version 3.3
September 2009

FINAL
QUALITY ASSURANCE PROJECT PLAN
FOR
REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT
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September 2009

APPROVAL PAGE

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9/1/2009

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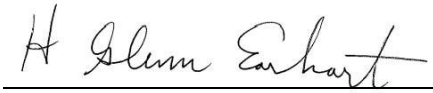

5/20/09

Glenn Earhart — Date
Program Manager

Richard Kennard — Date
USACE Louisville District Project Manager

Thomas Barounis — Date
US EPA Region 5 RPM

Charlene Falco — Date
Illinois EPA

Note: Regulatory Approvals may be in the form of concurrence letters, which will be included in the Final QAPP.

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LIST OF ACRONYMS

ADR	Automated Data Review
AES	Atomic Emission Spectroscopy
CD-ROM	Contract Disc-Read Only Memory
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COPC	Constituent of Potential Concern
DGM	Digital Geologic Map
DID	Data Item Description
DO	Dissolved Oxygen
DoD	Department of Defense
DOT	Department of Transportation
DQL	Data Quality Limit
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDS	Environmental Data Services, Inc.
EPA	United States Environmental Protection Agency
ERT	Earth Resources Technology, Inc
FS	Feasibility Study
ftp	File Transfer Protocol
FUDS	Formerly Used Defense Site
H2SO4	Sulfuric Acid
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochloric Acid
Hg	Mercury
HNO3	Nitric Acid
HTRW	Hazardous, Toxic and Radioactive Waste
ICP	Inductively Coupled Plasma
IEPA	Illinois Environmental Protection Agency
ITR	Internal Technical Review
J	Estimated
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Integrated Management System
LQAM	Laboratory Quality Assurance Manager
LSPM	Laboratory Services Project Manager
MDL	Minimum Detection Limit
MEC	Munitions and Explosives of Concern
mg/kg	milligrams per kilgram; parts per million
mg/L	milligram per liter; parts per million
mL	milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate

NaOH	Sodium Hydroxide
NELAP	National Environmental Laboratory Accredation Program
NPL	National Priority List
OB	Open Burn
OBG	Old Burning Ground
OD	Open Detonation
PARCCS	Precision, Accuracy, Representativeness, Completeness, and Comparability
pdf	Portable Document Format
PETN	Pentaerythritol Tetranitrate
pH	Potential of Hydrogen
PID	Photoionization Detector
PQL	Practical Quantitaion Limit
PQO	Project Quality Objective
QA	Quality Assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCP	Quality Control Plan
QL	Quantitation Limit
QSM	Quality System Manual
RCRA	Resource Conservation and Recovery Act
RDX	Cyclotrimethylenetrinitramine
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SCO	Site Cleanup Objective
SEDD	Staged Electronic Data Deliverable
SL	Screening Level
SOP	Standard Operating Procedure
SOW	Scope of Work
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
STR	Senior Technical Review
SUXOS	Senior Unexploded Ordnance Supervisor
SVOC	Semivolatile Organic Compound
SVAD	Savanna Army Depot Activity (used in conjunction with site designations)
SVDA	Savanna Army Depot Activity (used in conjunction with installation abbreviation only)
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TBC	To Be Considered
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound

TNT	Trinitrotoluene
TOC	Total Organic Carbon
TOX	Total Organic Halides
UFP	Uniform Federal Policy
ug/kg	Micrograms/kilogram; part per billion
ug/L	Microgram/Liter; part per billion
USACE	United States Army Corps of Engineers
UXO	Unexploded Ordnance
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
WC3	World Wide Web Consortium
WP	Work Plan
XML	eXtensible Markup Language
°C	Degress Celcius
%R	Percent Recovery

1.0 PROJECT MANAGEMENT AND OBJECTIVES

1.1 QAPP Identifying Information

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific quality assurance/quality control (QA/QC) procedures associated with the Remedial Investigation (RI)/Feasibility Study (FS) Work Plan (WP) for Site 50 (SVAD-050) at the Savanna Army Depot Activity (SVDA) in Savanna, Illinois.

The QAPP details specific protocols in place for field sampling, sample handling and storage, chain-of-custody, laboratory analysis, and data handling and management. The QAPP was prepared in accordance with *Uniform Federal Policy (UFP) for Quality Assurance Project Plans (QAPPs)* (EPA, December 2006) Table 1 provides a crosswalk of UFP required elements and where they apply in this QAPP.

Installation:	Savanna Army Depot (SVDA)
Operable Unit:	SVAD-050
*Contractor Name***:**	Earth Resources Technology, Inc. (ERT)
Contract Number:	W912QR-08-D-0012
Document Title:	Quality Assurance Project Plan (QAPP) for Remedial Investigation/Feasibility Study (RI/FS) at Open Burn/Open Detonation Area (SVAD-050) Savanna Army Depot
Work Assignment Number:	001
Regulatory program:	BRAC
Approval entity:	USACE, Louisville District
Scoping sessions held:	6 Sept 2008 and 8 Jan 2009
Data users:	USACE, SVDA
Excluded QAPP elements:	Tables/Worksheets omitted in instances where required information was repeated in other tables/worksheets or already present in text.

Part A of UFP QAPP Worksheet #2

Table 1. QAPP Elements and Corresponding QAPP Sections (UFP QAPP Worksheet #2)

Required QAPP Elements and Corresponding QAPP Sections	Required Information	Crosswalk to Related Documents
1.0 Project Management and Objectives		
Title and Approval Page	– Title and Approval Page	
Document Format and Table of Contents Document Control Format Document Control Numbering System Table of Contents 1.1 QAPP Identifying Information	– Table of Contents – QAPP Identifying Information	– Table 1
1.2 Distribution List and Project Personnel Sign-Off Sheet 1.2.1 Distribution List 1.2.2 Project Personnel Sign-Off Sheet	– Distribution List – Project Personnel Sign-Off Sheet	– Table 2 – Table 3
1.3 Project Organization 1.3.1 Project Organizational Chart 1.3.2 Communication Pathways 1.3.3 Personnel Responsibilities and Qualifications 1.3.4 Special Training Requirements and Certification	– Project Organizational Chart – Communication Pathways – Personnel Responsibilities and Qualifications Table – Special Personnel Training Requirements Table	– Figure 1 – Table 4 – QCP, RI/FS Work Plan, QAPP Attachment 2 – Table 5
1.4 Project Planning/Problem Definition 1.4.1 Project Planning (Scoping) 1.4.2 Problem Definition, Site History, and Background	– Project Planning Session Documentation (including Data Needs tables) – Project Scoping Session Participants Sheet – Problem Definition, Site History, and Background – Site Maps (historical and present)	– Tables 6 and 7 – Tables 6 and 7 – Table 8 – Section 3 of Sampling and Analysis Plan (SAP), Figures 2 and 3
1.5 Project Quality Objectives and Measurement Performance Criteria 1.5.1 Development of Project Quality Objectives Using the Systematic Planning Process 1.5.2 Measurement Performance Criteria	– Site-Specific PQOs – Measurement Performance Criteria Table	– Table 9 and Attachment 1 – Attachment 2, RI/FS Work Plan, GIP, Table 10
1.6 Secondary Data Evaluation	– Sources of Secondary Data and Information – Secondary Data Criteria and Limitations Table	– Work Plan section 1.8 – Table 11
1.7 Project Overview and Schedule 1.7.1 Project Overview 1.7.2 Project Schedule	– Summary of Project Tasks – Reference Limits and Evaluation Table – Project Schedule/Timeline Table	– Tables 12 through 18 – Attachment 1 – Figure 7

Table 1. QAPP Elements and Corresponding QAPP Sections (UFP QAPP Worksheet #2)

Required QAPP Elements and Corresponding QAPP Sections	Required Information	Crosswalk to Related Documents
2.0 Measurement/Data Acquisition		
2.1 Sampling Tasks 2.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 2.1.2.1 Sampling Collection Procedures 2.1.2.2 Sample Containers, Volume, and Preservation 2.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 2.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 2.1.2.5 Supply Inspection and Acceptance Procedures 2.1.2.6 Field Documentation Procedures	– Sampling Design and Rationale – Sample Location Map – Sampling Locations and Methods/ SOP Requirements Table – Analytical Methods/SOP Requirements Table – Field Quality Control Sample Summary Table – Sampling SOPs – Project Sampling SOP References Table – Field Equipment Calibration, Maintenance, Testing, and Inspection Table	– Appendix D (SAP) of WP – Figures 3-1, 3-2, and 3-3 of the SAP, Figures 4 through 6 – Table 19 – Table 21 – Table 22 – Attachment 3 – Table 20 – Table 23
2.2 Analytical Tasks 2.2.1 Analytical SOPs 2.2.2 Analytical Instrument Calibration Procedures 2.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 2.2.4 Analytical Supply Inspection and Acceptance Procedures	– Analytical SOPs – Analytical SOP References Table – Analytical Instrument Calibration Table – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	– Attachment 4 – Table 24 – Attachment 2 – Attachment 2
2.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 2.3.1 Sample Collection Documentation 2.3.2 Sample Handling and Tracking System 2.3.3 Sample Custody	– Sample Collection Documentation Handling, Tracking, and Custody SOPs – Sample Container Identification – Sample Handling Flow Diagram – Example Chain-of-Custody Form and Seal	– - Attachment 3 – Figure 8 – Table 25 – Attachment 2, Figure 9, and Attachment 1 of the SAP
2.4 Quality Control Samples 2.4.1 Sampling Quality Control Samples 2.4.2 Analytical Quality Control Samples	– QC Samples Table – Screening/Confirmatory Analysis Decision Tree	– Tables 10 and 22 – Not addressed

Table 1. QAPP Elements and Corresponding QAPP Sections (UFP QAPP Worksheet #2)

Required QAPP Elements and Corresponding QAPP Sections	Required Information	Crosswalk to Related Documents
2.5 Data Management Tasks 2.5.1 Project Documentation and Records 2.5.2 Data Package Deliverables 2.5.3 Data Reporting Formats 2.5.4 Data Handling and Management 2.5.5 Data Tracking and Control	– Project Documents and Records Table – Analytical Services Table – Data Management SOPs	– Table 26 – Not addressed – Not addressed
3.0 Assessment/Oversight		
3.1 Assessments and Response Actions 3.1.1 Planned Assessments 3.1.2 Assessment Findings and Corrective Action Responses	– Assessments and Response Actions – Planned Project Assessments Table – Audit Checklists – Assessment Findings and Corrective Action Responses Table	– – Table 27 – Attachment 5 –
3.2 QA Management Reports	– QA Management Reports Table	– Table 28
3.3 Final Project Report		–
4.0 Data Review		
4.1 Overview		
4.2 Data Review Steps 4.2.1 Step I: Verification 4.2.2 Step II: Validation 4.2.2.1 Step IIa Validation Activities 4.2.2.2 Step IIb Validation Activities 4.2.3 Step III: Usability Assessment 4.2.3.1 Data Limitations and Actions from Usability Assessment 4.2.3.2 Activities	– Verification (Step I) Process Table – Validation (Steps IIa and IIb) Process Table – Validation (Steps IIa and IIb) Summary Table – Usability Assessment	– Table 29 – Table 30 – Table 32 – Table 31 – Table 33
Streamlining Data Review Data Review Steps To Be Streamlined Criteria for Streamlining Data Review Amounts and Types of Data Appropriate for Streamlining	Not included	

1.2 Distribution List and Project Personnel Sign-Off Sheet

1.2.1 Distribution List

Table 2 provides contact information for the recipients of project planning documents and projects reports. The persons detailed in Table 2 are considered key personnel to the implementation and successful completion of the SVAD-050 RI/FS activities.

Table 2. Project Document Distribution List (UFP QAPP Worksheet #3)

Recipients	Organization/Title	Telephone Number	Email Address
Mr. John Clarke	Savanna Army Depot Activity (SVDA)	815-273-8827	John.E.Clarke@us.army.mil
Mr. Richard Kennard	USACE Louisville District Project Technical Manager	502-315-6323	Richard.A.Kennard@usace.army.mil
Mr. Glenn Earhart	ERT Program Manager	256-217-8991	glenn.earhart@ertcorp.com
Mr. Michael Dorman	ERT Project Manager	301-323-1401	mike.dorman@ertcorp.com
Mr. Thomas Barounis	US EPA Region 5 RPM	312-353-5577	Barounis.Thomas@epamail.epa.gov
Ms. Charlene Falco	Illinois EPA RPM	217-785-2891	Charlene.Falco@Illinois.gov

1.2.2 Project Personnel Sign-Off Sheet

Table 3 provides contact information for the personnel involved in preparing and implementing elements this QAPP for the SVAD-050 RI/FS activities.

Organization: Earth Resources Technology, Inc. (ERT)

Table 3. ERT Project Team Sign-Off Sheet (UFP QAPP Worksheet #4)

ERT Personnel	Title	Telephone Number	Email Address	Date QAPP Read
Mr. Glenn Earhart	ERT Program Manager	256-217-8991	glenn.earhart@ertcorp.com	
Mr. Michael Dorman	Project Manager	301-323-1401	mike.dorman@ertcorp.com	
Mr. Michael Garner	Senior UXO Supervisor/Site Manager	989-348-4189	ertci@I2k.com	
Mr. George Payne	UXO Safety Officer/UXO Quality Control Officer	301-323-1421	george.payne@ertcorp.com	
Dr. Wanfang Zhou	Technical Reviewer	256-217-8991	wanfang.zhou@ertcorp.com	
Ms. Monique Larriva	SAIC Project Manager	703-375-2046	monique.m.larriva@saic.com	
Mr. Pat Letterer	Project Manager, CT Laboratories, Inc.	800-228-3012	pletterer@ctlaboratories.com	
Mr. Douglas Weaver	Project Manager, Environmental Data Services, Inc.	757-564-0090	dweaver@env-data.com	

1.3 Project Organization

1.3.1 Project Organizational Chart

The primary personnel responsible for implementing the SOW are illustrated in Figure 1 below.

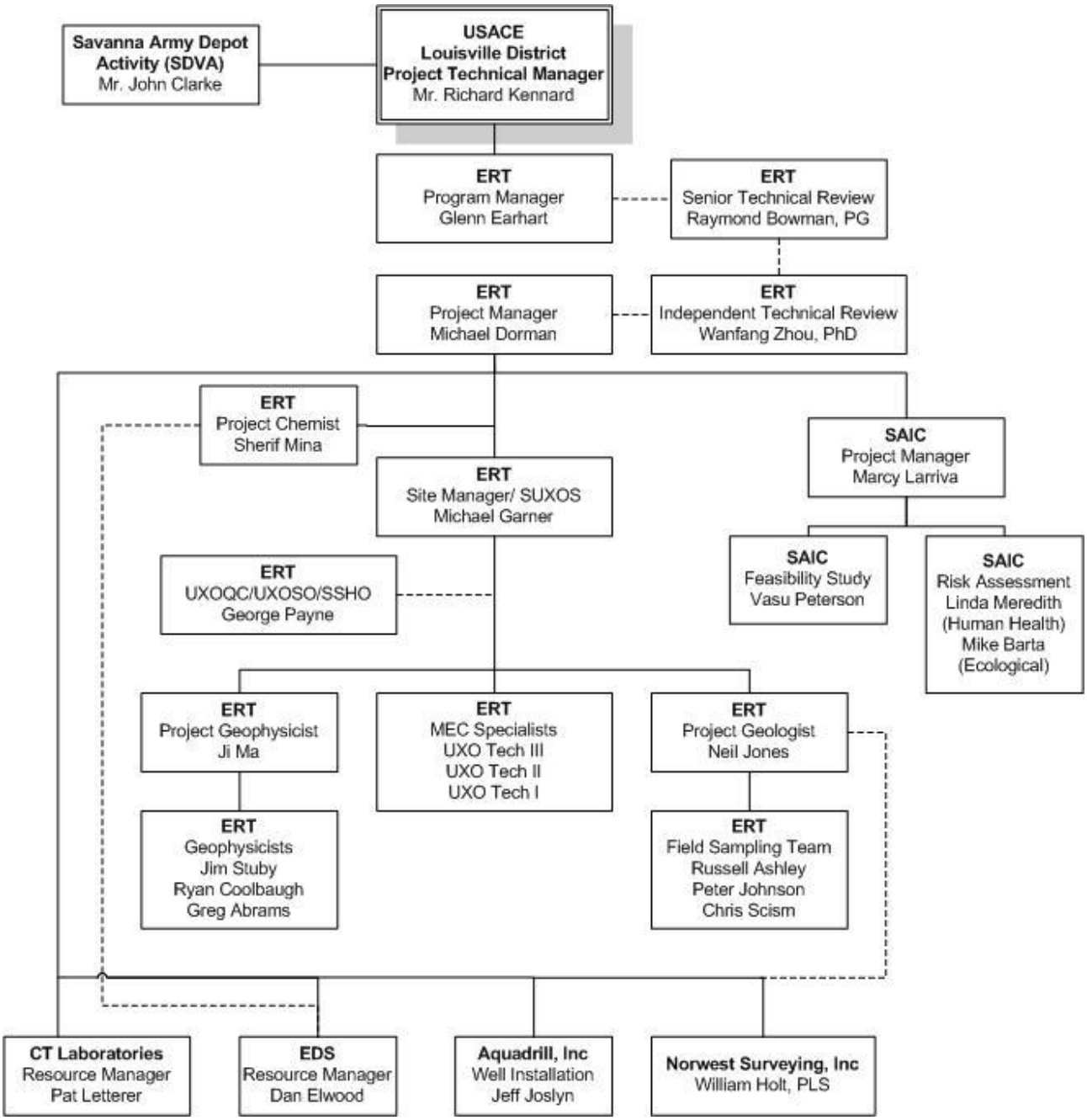


Figure 1. Project Organizational Chart

1.3.2 Communication Pathways

The ERT Project Manager will work in close communication with the USACE Louisville Project Technical Manager and the SVDA Base Realignment and Closure (BRAC) Environmental Coordinator (BEC) to ensure that all project requirements are met, and to keep the USACE Louisville Project Technical Manager informed of any technical or administrative issues that may impact the project schedule or budget. Any communication that has the potential to impact the project Scope of Work (SOW), schedule, or budget will be confirmed via written correspondence between ERT and the USACE Louisville Project Technical Manager.

Table 4. Communication Pathways (UFP QAPP Worksheet #6)

Communication Driver	Responsible Entity	Name	Telephone Number	Procedure
Savanna Army Depot Activity	BRAC Environmental Coordinator (BEC)	Mr. John Clarke	815-273-8827	All programmatic information, coordination issues, draft and final reports.
USACE Louisville District	Lead Technical Agency, Project Technical Manager (PTM)	Mr. Richard Kennard	502-315-6323	Procurement of service contracts and review and coordination of project plans and documents.
Contractor Program Lead	ERT Program Manager (PgM)	Mr. Glenn Earhart	256-217-8991	Coordinate ERT's programmatic direction with Louisville District and has ultimate responsibility for client satisfaction.
Overall Technical Lead	ERT Project Manager (PM)	Mr. Michael Dorman	301-323-1401	Manage schedule changes and funding needs, provide appropriate resources for execution of the SOW, develop project delivery team, and assist in design of the project with respect to overall objectives. Notify PTM and PgM of significant issues.
UXO Safety Lead	Site Manager (SM)	Mr. Michael Garner	989-348-4189	Communicate with PM and field personnel on safety issues, plan field activities safely and effectively.

Table 4. Communication Pathways (UFP QAPP Worksheet #6)

Communication Driver	Responsible Entity	Name	Telephone Number	Procedure
UXO Quality Control	UXO Safety Officer/UXO Quality Officer	Mr. George Payne	301-323-1421	Implementing SSHP requirements and conduct on-site safety training. Report any safety work stoppages immediately to the BEC and USACE. Report directly to SM and PM.
Geological Responsibilities	Project Geologist	Mr. Neil Jones	301-323-1407	Develop project planning documents, coordinate environmental sampling efforts with other field teams and the SM. Report to the ERT PM.
Geophysical Responsibilities	Project Geophysicist	Mr. Ji Ma	256-217-8991	Record and perform daily data review, processing, and dig-list picks. Report any problems and the solutions to the ERT PM.
QA/QC of Analytical data	Project Chemist	Mr. Sharif Mina	410-969-9080	Review analytical data and summarize the QC results for the ERT PM.
Risk Assessment Lead	SAIC Project Manager	Ms. Monique Larriva	703-375-2046	Perform a quality control review of the RI report, conduct the human health and ecological risk assessments, and prepare the FS. Report to the ERT PM.
Contracting Analytical Laboratory Responsibilities	CT Laboratories, Inc.	Mr. Pat Letterer	800-228-3012	Perform environmental sample analysis for both the quarterly groundwater monitoring and RI/FS components of this project. Report results to the ERT Project Chemist and EDS.
Analytical Data Validation Responsibilities	Environmental Data Services, Inc. (EDS)	Mr. Doug Weaver	757-564-0090	Conduct data validation of the laboratory analytical data. Report to the ERT Project Chemist.

1.3.3 Personnel Responsibilities and Qualifications

Presented this section are the individuals from ERT, the contract laboratory, and the data validation consultant that are responsible for the various QA elements of the program.

Earth Resources Technology, Inc. (ERT) will coordinate and manage the RI sampling and analysis program, data reduction, QA/QC, data validation, analysis, and reporting. ERT will direct the sampling activities, coordinate laboratory and field activities. A Senior Technical Review (STR) of all documents and phases of work will be conducted by the STR officer, Mr. Ray Bowman, P.G. Data validation services will be provided by Environmental Data Services (EDS).

The laboratory that will perform the analytical services, CT Laboratories, is a National Environmental Laboratory Accreditation Program (NELAP) certified laboratory. CT Laboratories will communicate directly with ERT regarding the analytical results, reporting of result and will be responsible for providing all labels, sample containers, field blank water, trip blanks, shipping coolers, and laboratory documentation.

Resumes for the ERT management team are provided in the Revised Final Quality Control Plan (QCP) (ERT, 2008). Additional key staff resumes for the delivery team are provided in the RI/FS Work Plan (ERT, 2009a).

Program Manager – Glenn Earhart

Mr. Glenn Earhart is responsible for contracting, oversees contract specifications to be included in the execution of the project task, and has ultimate responsibility for client satisfaction. Mr. Earhart has been the ERT Program Manager for USACE Hazardous, Toxic and Radioactive Waste (HTRW) projects for three years and has been managing USACE projects for over 20 years.

Project Manager – Michael Dorman

Mr. Michael Dorman is responsible for managing schedule changes and funding needs, providing appropriate resources for execution of the SOW, developing the project delivery team, assisting in the design of the project with respect to overall objectives, and managing the final STR and internal technical review (ITR). Mr. Dorman has over 30 years of experience and has served as a Deputy Program Manager and Project Manager on a number of USACE-Baltimore contracts.

Project Chemist – Sherif Mina

The Project Chemist, Mr. Sherif Mina, shall ensure that any necessary revisions are made and all project personnel, stakeholders, and subcontractors receive the revisions or revised versions of this QAPP. The Project Chemist shall check on the implementation of this QAPP during the life of the project. The Project Chemist shall ensure that all chemistry-related goals of the program are obtained. The Project Chemist shall also be responsible for coordinating communication between the project team and the subcontracted laboratory, and performing data verification and data review on the analytical data.

Technical Staff

A multidisciplinary team comprised of environmental engineers, civil engineers, environmental scientists, geologists, geophysicists, explosives experts, data coordinators, and information systems specialists has been developed to implement the project detailed in the SOW.

Quality Control Staff

Although the Program Manager, Project Manager, and Technical Site Manager will each be responsible for ensuring quality of project deliverables, specific personnel have been designated to provide senior and independent review of deliverables. Additional details of the project review process are included in the QCP (ERT, 2008).

Subcontractors

It will be necessary for services to be provided by subcontractors in order to complete the proposed activities for the RI. For a more detailed description of the anticipated services required, refer to the project QCP (ERT, 2008) and the SAP (ERT, 2009b). As the project proceeds ERT recognizes the potential for other subcontractors to be utilized in order to complete project objectives. As such occasions occur, the new subcontractors will be communicated to the USACE Louisville District Project Technical Manager or his/her designee.

Analytical Laboratories

All samples collected for laboratory analysis will be analyzed by:

CT Laboratories
1230 Lange Court
Baraboo, WI 53913
Phone: (608) 356-2760

Attachment 2 of this QAPP contains CT Laboratories Certifications, Corporate QA Manual (QAM), and the company DoD-Quality Systems Manual (QSM) Self-Certification Statement.

Laboratory Technical Director – Pat Lerrer

CT Laboratories will have a Technical Director responsible for overall technical operations. The Laboratory Technical Director, Mr. Pat Letterer, will have sufficient authority and be responsible for actively supporting the implementation of the Laboratory QAM, participating in proficiency testing, certifying personnel have appropriate education and training, maintaining adherence to established standard operating procedures (SOPs) and enforcing their use in the laboratory, ensuring adequate supervision of technical staff, providing a contingency plan, having policies and procedures in place that ensure protection of client's confidential information, and maintaining a work environment that emphasizes the importance of quality.

Laboratory Quality Assurance Manager

The Laboratory QA Manager (LQAM) will be responsible for maintaining the laboratory quality system and overseeing the QA aspects of the data. The LQAM shall develop, coordinate, and implement QA plans and procedures in support of the laboratory's projects. The LQAM shall also be responsible for monitoring the laboratory's activities for complying with this QAPP's policies and procedures, adhering to the contract scope of work, and implementing corrective actions for any QA/QC deficiencies.

The LQAM works independently of the Laboratory Technical Director and has "stop work" authority over all laboratory analyses. Additionally, the LQAM will certify that the data is in compliance with the terms and conditions of the ERT contract, scope of work, both technically and for completeness as required by the project. The LQAM will also authorize the release of the data contained in the hardcopy data package and in the computer-readable data.

Laboratory Services Project Manager

Functionally, reporting to the Project Chemist shall be the Laboratory Services Project Manager (LSPM). The LSPM, or designee, shall perform a final review of the data to determine if all analytical results of the samples are consistent. Correlation of results for different parameters of a sample is evaluated at this time before the data are presented in a final report to the client. If discrepancies or deficiencies exist in the analytical results, then corrective action is taken. The LSPM shall verify all environmental samples are analyzed for requested parameters, notify the Project Chemist of any laboratory nonconformances and provide laboratory results to ERT for the inclusion of data in project reports.

Laboratory Analysts

The laboratory analyst generates the data (i.e., logs in, prepares and/or runs the samples) and is responsible for primary review of those data. The primary review is often referred to as a "bench-level" review. One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project-specific requirements have been understood and followed. Once the analysis is complete, the primary reviewer ensures that sample preparation information is complete, accurate, and documented; calculations have been performed correctly; quantitation has been performed accurately; qualitative identifications are accurate; client-specific requirements have been followed; method and process SOPs have been followed; method QC criteria have been met; QC samples are within established limits; dilution factors are correctly recorded and applied; nonconformances and/or anomalous data have been properly documented and appropriately communicated (i.e. chromatograms before and after each manual integration and reason for manual integration are included in the data packages), and chain-of-custody procedures have been followed. If the instrument calibration and recoveries of all QC samples are within specified tolerances, then the data are presented for secondary review. If instrument calibration or the recoveries of any QC samples exceed specified tolerances, then affected sample results are evaluated and generally the samples are submitted for reanalysis. Any manual integration that occurs is dated and signed and, if appropriate, noted in the case narrative.

Data Validator – Nancy Weaver

All analytical data will be independently validated by Mrs. Nancy Weaver of:

Environmental Data Services, Inc. (EDS)
1156 Jamestown Rd., Suite A
Williamsburg, Virginia 23185
Telephone: 757-564-0090

Data validation will include reviews of the laboratory QA/QC procedures and results, and a review of the usability and defensibility of the data. Upon receipt of the data validation report,

the ERT Project Chemist will perform an internal verification of the reported data and data validation.

1.3.4 Special Personnel Training Requirements

Table 5. Site-Specific Training Requirements (UFP QAPP Worksheet #8)

Team Member	Site-Specific Training	Medical Surveillance	Training Requirements
Site Manager	Yes	Yes	40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) 8-Hour HAZWOPER Site Supervisor CPR
Site Safety and Health Officer (SSHO)	Yes	Yes	40-Hour HAZWOPER CPR
Senior Unexploded Ordnance Supervisor (SUXOS)	Yes	Yes	40-Hour HAZWOPER Further detailed in the Munitions and Explosives of Concern (MEC) Investigation Plan
Unexploded Ordnance (UXO) Personnel	Yes	Yes	40-Hour HAZWOPER Further detailed in the MEC Investigation Plan
Environmental Field Technician	Yes	Yes	40-Hour HAZWOPER
Field Personnel	Yes	Yes	40-Hour HAZWOPER

1.4 Project Planning/Problem Definition

1.4.1 Project Planning (Scoping)

Two project scoping sessions were conducted in preparation for RI/FS activities at SVAD-050. Provided below are the project scoping session participant sheets created for each meeting (see Tables 6 and 7).

Table 6. Project Scoping Session #1 (UFP QAPP Worksheet #9)

Title: Site Visit
Revision No: 1
Revision Date: 1/12/2009
Page 1 of 1

Project Scoping Session Participants Sheet

Project Name	SVAD-050 RI/FS	**Site Name**	SVAD-050
Projected Sampling Date	Spring 2009	**Site Location**	Savanna, Illinois
Project Manager	Mike Dorman		
Date of Session:	9/6/2008		
Purpose of Session:	Site Visit		

Name	Title	Affiliation	Ph#	Email	Role
John Clarke	BRAC Env. Coordinator	US Army	(815)273-8827	john.e.clarke@us.army.mil	Oversight, Review
Glenn Earhart	ERT Program Manager, Huntsville Office	ERT	(256) 679-1070	glenn.earhart@ertcorp.com	Coordinator
Mike Garner	Site Manager	ERT	(256) 217-8991	ertci@i2k.com	SUXO
Neil Jones	Project Geologist	ERT	(301) 323-1407	neil.jones@ertcorp.com	Geologist

Discussion: Identified the Open Burn and Open Detonation Areas, Located several wells, inspected berms in Open Detonation areas. Looked at available storage/field offices.

Action Items: Contact USACE, Louisville about previous investigations, official site boundaries and gathering any other relevant documentation.

Table 7. Project Scoping Session #2 (UFP QAPP Worksheet #9)

Title: Kick-Off Meeting
Revision No: 1
Revision Date: 1/15/2009
Page 1 of 1

Project Scoping Session Participants Sheet

Project Name	SVAD-050 RI/FS	**Site Name**	SVAD-050
Projected Sampling Date	Spring 2009	**Site Location**	Savanna, Illinois
Project Manager	Mike Dorman		
Date of Session:	1/8/09		
Purpose of Session:	Kick-Off Meeting		

Name	Title	Affiliation	Ph#	Email	Role
Richard Kennard	Project Geologist	USACE-LRL	502-315-6323	Richard.A.Kennard@usace.army.mil	Project Technical Manager
Karen Rabek	Project Geologist	USACE-LRL	502-315-6328	Karen.V.Rabek@usace.army.mil	Project Geologist
Michael Dorman	Regional Manager	ERT	301-323-1401	michael.dorman@ertcorp.com	Project Manager
Glenn Earhart	Program Manager	ERT	256-217-8991	glenn.earhart@ertcorp.com	Program Manager

Discussion: Discussion focused on the new site boundary provided by John Clarke at the installation and EPA Region 5 preference for putting the QAPP in the UFP format. Because of the new site boundary, some of the field activities will be less extensive, but the planning documents will now require 5 versions, vs the 3 versions originally planned.

Action Items: ERT will update all Draft Final Plans with the new site boundary and put the QAPP into UFP format. Plans will be submitted to USACE-LRL and SVDA for review. Once plans have been submitted, ERT will revise the cost estimate to incorporate the new site boundary and increased project plan effort. Costs will not exceed current funding levels.

1.4.2 Problem Definition, Site History, and Background

Table 8. Problem Definition (UFP QAPP Worksheet #10)

The problem to be addressed by the project

RI/FS activities at SVAD-050 are being performed under the Department of the Army Base Realignment and Closure (BRAC) program. A preliminary review of the site and previous investigations indicates that soil, groundwater, surface water, and sediment are potentially affected by historical SVAD-050 activities. It is not clear, however, whether the source-receptor interaction occurs at concentrations that may pose an unacceptable risk to human health and the ecological environment. ERT will collect data necessary to identify environmental impacts associated with former DoD activities, including the use of the open burn (OB) and open detonation (OD) sites. Data collected will be used to help determine the extent in which future RI activities should proceed at SVAD-050, and provide a comparison of the RI/FS results to future remedial actions to site-specific cleanup objectives (SCOs).

The environmental question to be asked

1. What are the Chemicals of Potential Concern (COPC) at SVAD-050, and do they present an unacceptable risk to human health and the environment as compared to:

Screening of chemical and munitions constituent analytical data will be performed using the most recent IEPA Tiered Approach to Corrective Action Objectives (TACO) Tier I values and USEPA Regional Screening Levels (RSLs). Updates to Illinois groundwater quality standards and TACO objectives will be included as screening criteria, should they become available before the investigation is completed.

Soil

- USEPA Sept 2008 Screening Levels (SLs) for Residential Soil
- Illinois Tier 1 Soil Remediation Objectives for Residential Properties
- USEPA Region 5 Ecological Screening

Groundwater (GW)

- USEPA Sept 2008 Tapwater SLs
- USEPA Sept 2008 Maximum Contaminant Limits (MCLs)
- IEPA GW Quality Standards
- USEPA Region 5 Ecological Screening

Surface Water (SW)

- IEPA SW Quality Standards for human health and chronic aquatic life criteria (or Illionois' surface water criteria for chemicals not listed in the SW Quality Standards)
- USEPA Region 5 Ecological Screening

Sediment (To be considered (TBC))

- Illinois Sieved Stream Sediment Data, National Sediment Quality Survey, and/or USEPA Sept 2008 Residential SSLs.
- USEPA Region 5 Ecological Screening

Table 8. Problem Definition (UFP QAPP Worksheet #10)

2. What is the vertical and horizontal extent of any potential contamination?

Soil

- Surface soils will be field screened for explosives following a systematic grid system approach (88 locations) along with 17 locations to be determined in the field.
- Anomalies identified by geophysical surveys and investigated by digging up to 6 test pits. Two soil samples will be collected at each test pit: one at the surface (prior to excavation) and one at the bottom of the pit.
- Twenty five surface and 25 subsurface soil samples collected.

Groundwater

- New groundwater well installed in the center of the OD area.
- Existing monitoring well data to be used.
- Perchlorate will be sampled at all well in conjunction with the fourth quarter 2009 groundwater monitoring program

Sediment

- Ten surface sediment samples collected (0-6 inches below ground surface)
- Five subsurface sediment samples collected (2-3 feet below ground surface)

Surface Water

- Five surface water samples collected around the perimeter of the site.

Observations from any site reconnaissance reports

A site visit to SVDA during scoping sessions (see Tables 6 and 7) included the collection of site specific operations information and a visual survey of SVAD-050. The visual survey was to identify and inspect existing wells, berms, boundaries, topography, and surface water conditions. A second scoping session took place in January 2009 to clarify the boundary of the study area.

Based on observations from the visual survey, it was determined that SVAD-050 consists of land with multiple man-made features elevated above surface water in the form of Mississippi River backwater/wetlands. Discussions concluded that the SVAD-050 site has been flooded multiple times since 1950, and was completely submerged in 2001. Tentative surface water sampling locations were identified to be equally spaced around the perimeter of SVAD-050.

Nine groundwater monitoring wells currently exist along the boundaries of SVAD-050, and are currently active for a Resource Conservation and Recovery Act (RCRA) quarterly groundwater monitoring activities. Proposed placement location of an additional monitoring well in the center of the OD Area within SVAD-050 was identified.

Elevated burn pads were identified as potential soil sample locations.

A synopsis of secondary data or information from site reports

There have been a number of additional investigations in and around SVAD-050 over the past 30 years that have confirmed the presence of site-related chemical constituents in the environment. These studies are identified and summarized in Section 1.8 of the RI/FS Work Plan (ERT, 2009a), and are included in Table 11, Secondary Data Criteria and Limitations.

Table 8. Problem Definition (UFP QAPP Worksheet #10)

The possible classes of contaminants and the affected matrices

A full suite of analytes to be examined during the initial Phase I soil sampling in and around SVAD-050 includes: Target Compound List (TCL) volatile organic compounds (VOCs), TCL semi-volatile organic compounds (SVOCs), Target Analyte List (TAL) metals, pesticides, polychlorinated biphenyls (PCBs), cyanide, perchlorate and explosives. Based on the results of the Phase I samples, some methods may be dropped during Phase II sampling if no analytes were detected above screening levels (or not detected at all). The Phase II effort will include at least 50 additional soil samples (25 surface and 25 subsurface), groundwater, surface water and sediment samples and will include a reduced analytical suite, plus perchlorates in groundwater and nitrate/nitrite in aqueous samples. This analysis suite was chosen to provide a comprehensive look at the contaminants possibly present at SVAD-050. It is anticipated that the most likely and abundant classes of contaminants present will be, metals, and explosives in all matrices (and possibly some VOCs, SVOCs).

The rationale for inclusion of chemical and non-chemical analyses

The nature of activities within and around SVAD-050 provides the basis for selecting the analytical suite to be examined. Historically, SVAD-050 was used by the Department of the Army since 1930s for the burning of unstable propellants (OB area), demolition of various explosives by detonation (OD area), and transport (along the gravel road) of munitions, explosives, and waste to and from the site.

The OB sites consisted of metal pans elevated above concrete pads by steel beams used to burn propellants, thereby releasing potentially releasing propellants.

The OD grounds within SVAD-050 were used to detonate obsolete projectiles and ordnance. Example explosives used in detonation/demolition activities included: trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), and pentaerythritol tetranitrate (PETN). The OD area consisted of two long earthen berms where pits were located, three pits per berm. Obsolete projectiles were destroyed energetically with a series of "shots." After the series of "shots" the surrounding area was searched for unexploded material. Materials that were not sufficiently destroyed were either collected and prepared for the next series of shots or detonated in place. Debris from demolition has spread across the site and into the surrounding water, sediment, and bottomlands around the burning pits, thereby potentially spreading explosive and metals constituents across the site.

SDVA is a National Priority List (NPL) installation with RCRA interim status, extended closure period site (IL3210020803 - also known as the OB/OD Area or SVAD-050). Adjacent to SVAD-050 is a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) site known as the Old Burning Ground Group (OBG or SVAD-009-R-01). Similar historical activities were conducted at both sites. However, the OBG has previously been investigated in adherence to CERCLA regulation and standards, including a comprehensive RI and subsequent addendums.

There have been a number of additional investigations in and around SVAD-050 over the past 30 years which help provide the rationale for inclusion of the chemical and non-chemical measures

Table 8. Problem Definition (UFP QAPP Worksheet #10)

of this RI/FS. These studies are identified and summarized in the Table 11.

Information concerning various environmental indicators

Water surrounds SVAD-050, and includes portions of Crooked and Straight Sloughs, and backwater areas of the Mississippi River within Pool 13, south of L&D No. 12. In general, the backwater areas within Pool 13 consist of a series of shallow sloughs and oxbows with silt and sand substrates, often in layers at least 2 ft deep; they receive water from offshoot channels of the Mississippi River and from seasonal flooding. The rising and falling water levels within the pool alter water depth, sediment transport, and general water quality. Unlike the main river channel, water movement in the sloughs is usually minimal.

The shallow aquifer underlying the site area consists of fine- to coarse-grained sand with interlayered clay (Dames & Moore, 1994), with water levels below land surface (bls) typically less than 10 feet.

Figure 2 presents the SDVA location map, and illustrates the installations proximity to the Mississippi River and the Mississippi River backwater and wetlands. Figure 3 presents the SVAD-050 Site Map, and illustrates the site boundaries, proximity to the Mississippi River and the Mississippi River backwater and wetlands.

Project decision conditions

If analytes within the target analyte groups are detected at concentrations meeting or exceeding the relevant USEPA or Illinois (TACO) Screening Levels, then the analytes will be categorized as a COPC.

If COPC are identified, then these data will be used to help determine the extent in which future RI activities should proceed at SVAD-050, and provide a comparison of the RI/FS results to future remedial actions to site-specific cleanup objectives (SCOs).

Figure 2. Savanna Army Depot Location Map

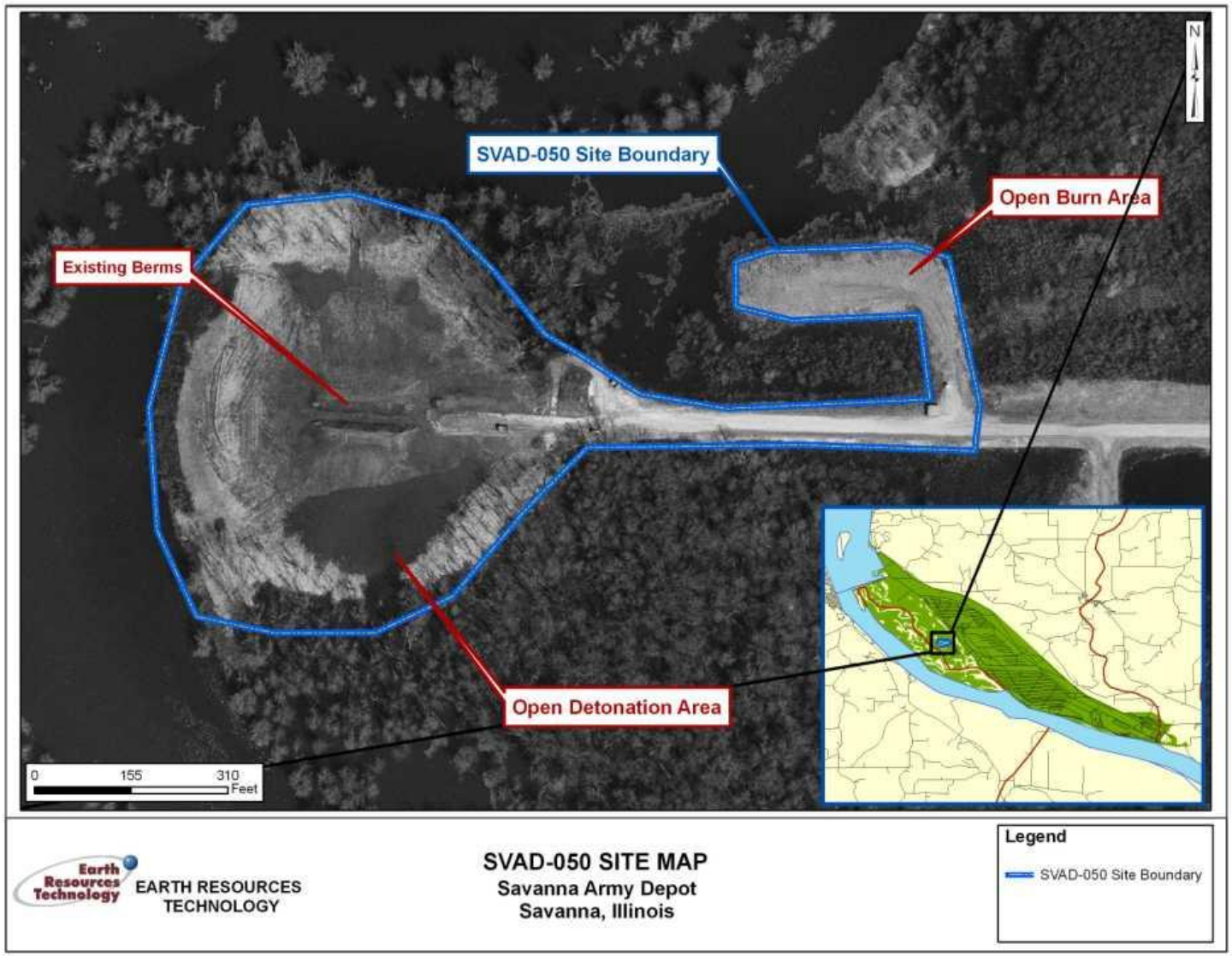


Figure 3. SVAD-050 Site Map

1.5 Project Quality Objectives and Measurement Performance Criteria

1.5.1 Development of Project Quality Objectives Using the Systematic Planning Process

The Data Quality Objectives (DQO) process flow for SVAD-050 follows the requirements of EPA QA/G-4 (EPA, 2006).

Step 1. State the problem

- What is the extent of lateral and vertical contamination at SVAD-050?
- What risks, to both human and ecological receptors, posed by potential contamination at the site?
- If risks are present, what remedial alternatives are necessary to reduce risks to acceptable levels?

Step 2. Identify the decision

- Analytical sampling, field screening and digital geophysical mapping (DGM) results will be used to determine if COPC and MEC are present.
- Results will determine if COPC and MEC represent a risk to human health or the environment.
- Results will delineate the extent of the COPC.
- Results will determine the level of remediation necessary to reduce risk to acceptable levels.

Step 3. Identify the inputs to the decision

- Previous investigation reports.
- Analytical results (see Attachment 1 for a complete list of analytes) and field screening results.
- DGM results
- USEPA Screening Levels (SLs) for Tapwater (GW/SW) and Residential Soil (Soil/Sediment), Illinois Water Quality Standards (GW/SW), and Illinois Tier 1 Soil Remediation Objectives (see Attachment 1 for a complete list of SLs).
- Sample location, type, and depth.
- Future land use requirements.
- Applicable or Relevant and Appropriate Requirements.

Step 4. Define the study boundaries

- Confined to the vicinity of SVAD-050.
- Confined to shallow groundwater, surface and subsurface soil and sediment, and surface water.

Step 5. Develop a decision rule

- Gain acceptance of spatial extent of soil sampling plan by developing a technical memo to identify proposed sampling locations based on field screening, anomaly identification, and test pits results for review by IEPA

- If analytical data results exceed USEPA Regional or Illinois Screening Levels, then additional evaluations of potential human health and environmental risks may be warranted.
- If risks, to human or ecological receptors, are unacceptable, an evaluation of remedial alternatives will be performed to reduce those risks to acceptable levels.

Step 6. Specify tolerable limits on decision error

- Quantitative project specific objectives for the data quality indicators of precision, accuracy, completeness and sensitivity have been developed to define acceptable measurement error.

Step 7. Optimize the design

- This QAPP was developed based on the needs of the project and obtaining sufficient quality data to address the project objective.

The project quality objectives for the RI/FS at SVAD-050 are presented in Table 9.

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

Who will use the data?

USACE, USEPA, SVAD, Illinois EPA, ERT, SAIC

What will the data be used for?

Data will be used to:

- Ensure a safe working environment for field personnel conducting RI activities.
- Utilize systematic and biased sample locations which adequately provide spatial coverage for SVAD-050.
- Identify COPC.
- Determine the extent in which future RI activities should proceed at SVAD-050.
- Provide a comparison of the RI/FS results to future remedial actions to site-specific cleanup objectives (SCOs).

What type of data is needed?

Overall

- A geophysical investigation is necessary to provide MEC clearance.
- Previous sampling data (i.e. quarterly RCRA sampling data) is necessary to fully characterize SVAD-050.
- Analysis of TCL VOCs, TCL SVOCs, TAL metals, pesticides, polychlorinated biphenyls (PCB), cyanide, anions, perchlorate, and explosives by USEPA SW 846 Methods is necessary to characterize soil, groundwater, surface water, and sediment conditions (see Attachment 1 for methods).
- Environmental data must be screened versus the following:
    - Soil results will be screened against IEPA Tier 1 Soil Remediation Objectives for Residential Properties and USEPA Sept 2008 RSLs for Residential Soil.
    - Groundwater results will be screened against IEPA GW Quality Standards, USEPA Sept 2008 Tapwater RSLs, and USEPA MCLs.
    - Surface water results will be screened against IEPA SW Quality Standards for human health, and USEPA Sept 2008 Tapwater RSLs.
    - Sediment will be considered to be screened against Illinois Sieved Stream Sediment Data, National Sediment Quality Survey, and/or USEPA Sept 2008 Residential RSLs for Residential Soil.
    - Ecological screening will be based upon USEPA Region 5 Ecological Screening Levels and Illinois Surface Water Quality Standards for Aquatic Life (chronic).
- The Reference Limits and Evaluations criteria (Attachment 1) are needed to evaluate the data. ERT has incorporated USEPA and IEPA screening levels, and chosen the most

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

sensitive analytical methods with reporting limits (RLs) that are lower than the data quantitation limit (DQL, screening level) values wherever possible. For those parameters whose RLs are still greater than the associated DQL values, there is no practical, alternative method that could provide a lower reporting limit. Those compounds are usually carried forward to the risk assessment. Because these compounds are as critical as other compounds with associated screening levels, this practice will be likely to err on the side of health protection (or false positive) in the risk assessment.

- For analytes with no specified DQL (listed as NS), they will be referenced but will not be used directly for risk assessment.
- Phase I soil sampling consists of laboratory analysis for a minimum of two soil samples (one surface and one subsurface) collected from each test pit, and surface soil field screening samples collected based upon a pre-established grid system. After a review of the DGM investigations, field screening soil sample results and Phase I soil sample results, a second phase of surface and subsurface soil sampling will be conducted.
- Phase II soil sampling will consist of surface and subsurface samples collected for laboratory analysis from 25 locations. Some of the Phase II soil samples will be analyzed for TCLP analytes (up to 4 samples). The Phase II sampling will also include groundwater sampling of one new monitoring well, surface water sampling and sediment sampling. Surface water and sediment samples will be collected from a pond within the OD area and along the banks of open waterways adjacent to SVAD-050.

Soil

- Field screening for explosives (using DropEx®), visual observations, and screening for volatile organics by PID measurements is necessary to identify areas most likely impacted and identify soil sampling locations.
- Phase I soil sample shall be collected from within test pits at locations adjacent to items found in the pit soils.
- Phase II soil samples shall be collected from the subsurface at the area of most likely impacted by examining all subsurface intervals (in the unsaturated soil extending from the ground surface to the water table) by visual inspection and PID measurements run over the length of the soil boring. The Phase II samples will also be collected to ensure the boundaries of any contamination are determined (so some samples may be collected in areas expected to be uncontaminated).
- Phase II soil samples shall be collected from the surface (from 0-6 inches below ground surface (bgs)) at locations determined by the explosives field screening during the Phase I field activities, the test pits analytical results and the DGM.
- Sampling by hand auguring is the preferred field method for the collection of Phase II subsurface soil samples.

Groundwater

- One new groundwater monitoring well will be installed in the center of the berm in the detonation area.
- Groundwater sampling will follow low-flow methodology.

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

- Water quality parameters (pH, temperature, turbidity, conductivity, dissolved oxygen, and ORP) are necessary to monitor for groundwater stabilization prior to sample collection.
- Samples will be analyzed for full suite analysis (unless Phase II results show no particulat analyte groups are present), plus perchlorate and nitrate/nitrite.

Surface Water

- Samples will be collected from within standing water greater than 12 inches in depth, during baseline conditions (when flooding is not occurring).

Sediment

- At a minimum, sediment samples will be collected from within standing water greater than 12 inches in depth. For the purpose of the RI, sediment will be considered soils present below the mean water level of any static or open water body that is saturated greater than 50% of the year. This definition is intended to exclude soils which lie at the base of puddles, drainage ditches, swales and other similar formations. Mean water level will be established by visual observation of shoreline water marks and/or by interviews with on-site personnel.
- Surface sediment samples will be taken from 0 - 6 inches bls. Subsurface sediment samples will be taken between 2 and 3 feet bls.
- Sediment samples will be treated as soil in the laboratory and corrected for dry weight. It should be expected that RLs and minimum detection limits (MDLs) will increase accordingly.

Data Quality

The quality of the data will be determined for performance measures; including precision, accuracy/bias, sensitivity (quantitation limits), data comparability, representativeness, and completeness.

- Analytical precision will be determined predominantly by the collection and analysis of field duplicate samples and by analysis of laboratory matrix spikes/matrix spike duplicates (MS/MSD). Precision will be measured through the calculation of Relative Percent Difference (RPD) as follows:

$$RPD = \left| \frac{A - B}{mean} \right| \times 100$$

  Attachment 2 summarizes the acceptable limits of laboratory precision per method and analyte.

- Accuracy/bias will be ensured through the adherence to all field instrument calibration procedures; sample handling, preservation, and holding time requirements; and through the collection of rinsate blanks prior to the collection of samples for each type of

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

equipment being used (e.g., split spoons, groundwater sampling pumps).

The laboratory will assess the overall accuracy of their instruments and analytical methods (independent of sample or matrix effects) through the measurement of "standards," materials of accepted reference value. Accuracy may vary from analysis to analysis because of individual sample and matrix effects Accuracy within each individual analysis will be measured in terms of blank results, the recovery of surrogate compounds in organic analyses, or percent recovery (%R) of spiked compounds in MSs, MSDs and/or Laboratory Control Samples (LCSs). This gives an indication of expected recovery for analytes tending to behave chemically like the spike or surrogate compounds.

Cumulative laboratory and method accuracy/bias will be calculated as a percentage as follows:

$$\text{Accuracny/Bias} = \frac{Actual}{Theoretical} \times 100$$

Spiked recoveries, expressed as % R, will be calculated as follows:

$$\%\text{R} = \frac{SpikedSampleConc - UnspikedSampleConc}{SpikeConcAdded} \times 100$$

Measurement Performance Criteria (MPC) for precision and accuracy measures is presented for the analyte groups in the CT Laboratories QA Manual, and exact MPC data for specific analytes are presented in the CT Laboratories QA/QC Limits Document (see Attachment 2).

- The sensitivity of most laboratory MDLs are below the relevant USEPA Screening Levels. MDLs are presented per analyte and matrix in Attachment 1.

- All field activities will be conducted following a set of ERT SOPs (see Attachment 3). Analytical data sets will be generated following standardized methods and Laboratory SOPs (see Attachment 4) and by using traceable standards. Data will be reported in consistent dry weight units for solid samples (i.e., µg per kg and/or mg per kg, and µg per liter (L) or mg/L for aqueous samples). Comparability with historic data sets will be evaluated based on similarities or differences between sampling design and field and analytical protocols.

- To ensure representativeness, the sampling locations have been selected to provide coverage over a wide area and to highlight potential trends in the data. Methodologies found in the SVAD-050 RI/FS Work Plan and this QAPP will be followed to ensure that proper sampling technique, sample management, and sample preservation

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

techniques are followed.

Representativeness in the laboratory will be ensured by proper analytical procedure, appropriate methodology, and meeting sample holding times.

- The field completeness objective (a measure (in percent) of the amount of valid measurements obtained compared to the amount of measurements expected to be taken in the project) is greater than 90 percent. The laboratory completeness objective for the amount of valid measurements obtained per analyte compared to the amount expected per analyte is 95 percent.

$$\%\,\text{Complete} = \frac{Number of Valid Measurements}{Number of Expected Measurements} \times 100$$

How much data are needed?

See Table 10 and next section.

Where, when, and how should the data be collected/generated?

Soil

Phase I soil sampling will consist of roughly 105 field screening soil samples and 12 soil samples collected for laboratory analysis from the selected test pit locations. At the conclusion of Phase I sampling, the results (both field screen and laboratory analytics) will be reviewed. Based upon this data and an evaluation of geophysical investigation results, a minimum of 25 sample locations will be selected for Phase II soil sampling, as areas of further investigation.

- A systematic sampling approach has been designed to field screen soil samples, in the entire SVAD-050 area (see Figure 4). Sample locations will be located on 50 ft or 100 ft centers, with the exception of where sample locations encounter former structures that are unable to be penetrated. Systematic samples originally placed in such described locations will be offset a minimal distance to remove the obstruction from the sample location. A total of 88 systematic field screening sample locations, based on grid nodes, and 17 additional biased field screening samples will be collected based on field observations during Phase I. These surface soil samples are to be field screened for explosives (using DropEx® explosives detection kit)

- Based upon the analysis of the geophysical investigation, six test pit excavations will be excavated. As described in the GIP, criteria for selecting test pit excavation locations will be based on factors such as high concentrations of geophysical anomalies. A minimum of one test pit will be excavated at each of the OD and OB areas. Within each excavated test pit, a minimum of two soil samples, surface and subsurface, will be collected for laboratory analysis.

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

- Soil samples at the test pits will be collected as follows:
  - The surface soil sample will be taken at the center of the area to be excavated, using the procedures described below;
  - The subsurface soil sample will be taken immediately adjacent to any objects found within the test pit.

- After a review of the geophysical investigations and field screening soil sample results (Phase I soil sample results), a second phase of surface and subsurface soil sampling will be conducted. Phase II soil sampling will consist of surface and subsurface samples collected for laboratory analysis from at least 25 locations. The Phase II sampling will also include sampling of groundwater from the new monitoring well (described below), surface water, and sediment.

- Two soil samples will be collected from each Phase II sample location for laboratory analysis. One sample, a surface soil sample, will be collected from 0-6 inches below ground surface (bgs). The second soil sample will be collected from the area of most likely impact.

- PID measurements and observation of staining/discoloration will be conducted in all segments of each subsurface soil boring to determine exact sampling location. Soil samples will be collected where PID readings are highest and discoloration/staining is most obvious. This allows for identification and sampling of soil where concentrations of contaminants are potentially the highest.

Groundwater

One groundwater well will be installed and sampled in the center of the berms of the OD area (see Figure 5). The results of this groundwater sample will be assessed with groundwater data generated from quarterly RCRA sampling events at nine wells within SVAD-050, to identify COPC and evaluate potential impacts to groundwater.

Surface Water

Surface water samples will be collected; one from within the pond located in the OD area and four from the open waterway bordering SVAD-050. One of the open waterway samples will be collected immediately to the west of the OD Area, one immediately to the north of the OD Area, one between the OD and OB Areas, and one immediately to the north of the OB Area. The approximate proposed sample locations are presented in Figure 6. The exact location of these samples is unknown and will be predicated upon site condition during field activities. Seasonal flooding occurs at the site and greatly impacts potential sampling locations. At a minimum, surface water samples will be collected from within standing water greater than 12 inches in depth.

Sediment

Surface sediment samples will be collected at the same locations where surface water samples

Table 9. Project Quality Objectives/Systematic Planning Process

(UFP QAPP Worksheet #11)

are collected. Subsurface sediment samples (2-3 feet bgs) will also be collected where the surface water and surface sediment samples are collected. An additional five surface sediment samples will be collected adjacent to the surface water and sediment samples, as presented in Figure 6. The exact location of these samples is unknown and will be predicated upon site condition during field activities. Seasonal flooding occurs at the site and greatly impacts potential sampling locations. For the purpose of the RI, sediment will be considered soils present below the mean water level of any static or open water body that is saturated greater than 50% of the year. This definition is intended to exclude soils which lie at the base of puddles, drainage ditches, swales and other similar formations. Mean water level will be established by visual observation of shoreline water marks and/or by interviews with on-site personnel.

Field work for the SVAD-050 RI/FS will be conducted during the Summer 2009, with data evaluation and reporting occurring in Fall 2009. All work performed will be of sufficient quality to meet the requirements of *40 Code of Federal Regulation (CFR) 300.430* (EPA, 1988).

Who will collect and generate the data?

- Earth Resources Technology, Inc. (ERT) of Annapolis, Maryland will conduct all field activities.
- CT Laboratories, Inc. of Baraboo, Wisconsin will analyze all environmental samples.
- Environmental Data Services, Inc. (EDS) of Williamsburg, Virginia will perform all data validation.

How will the data be reported?

A compact disc-read only memory (CD-ROM) containing Adobe Portable Document Format (pdf) of the complete analytical data package including all raw data will be submitted by CT Laboratories to ERT.

CT Laboratories will prepare and submit USEPA CLP-like Level IV (Level D) data packages and electronic data deliverables (EDDs) to include a cover sheet, table of contents, case narrative, the analytical results, sample receipt and data management records, internal laboratory QA/QC information, and raw data. The laboratory data package should be organized so that the analytical results are reported on a per batch basis. The approved laboratory will also provide the chemical analysis results as electronic data deliverable (EDDs). For the purpose of having a uniform delivery format for analytical data from laboratories performing environmental analyses, and to ensure that this data can be reviewed in a timely manner prior to use, the USACE (under the FUDS Program) requires implementation of the Staged Electronic Data Deliverable/ Automated Data Review (SEDD/ADR) process.

How will the data be archived?

Hard copies and digital information produced by this investigation will be kept and organized by the ERT Project Manager at ERT's Annapolis Junction office for at least five years. All records will be offered to Mr. Thomas Barounis of USEPA Region 5 prior to disposal.

Table 10. Summary of Proposed RI/FS Samples at SVAD-050

Sample Type	Matrix	Phase of Sampling	Total Number of Samples	Surface Samples	Subsurface Samples	Duplicate Samples	ERB Samples	FB Samples	MS/MSD Samples
DropEx® Soil Screening	Soil	Phase I	116	105	0	11	0	0	0
Test Pit (Soil)	Soil	Phase I	15	6	6	1	1	0	1
Biased	Soil	Phase II	59	25	25	5	2	0	2
Sediment	Sediment	Phase II	19	10	5	2	1	0	1
Groundwater[1]	Aqueous	Phase II	3	N/A	1	1	0	0	1
Surface Water	Aqueous	Phase II	9	5	N/A	1	1	1	1

ERB Equipment Rinsate Blank
FB Field Blank
MS/MSD Matrix spike/Matrix spike duplicate
[1] All groundwater and associated QC samples from MW-11 will be sampled in conjunction with regular RCRA sampling at the OB/OD Areas

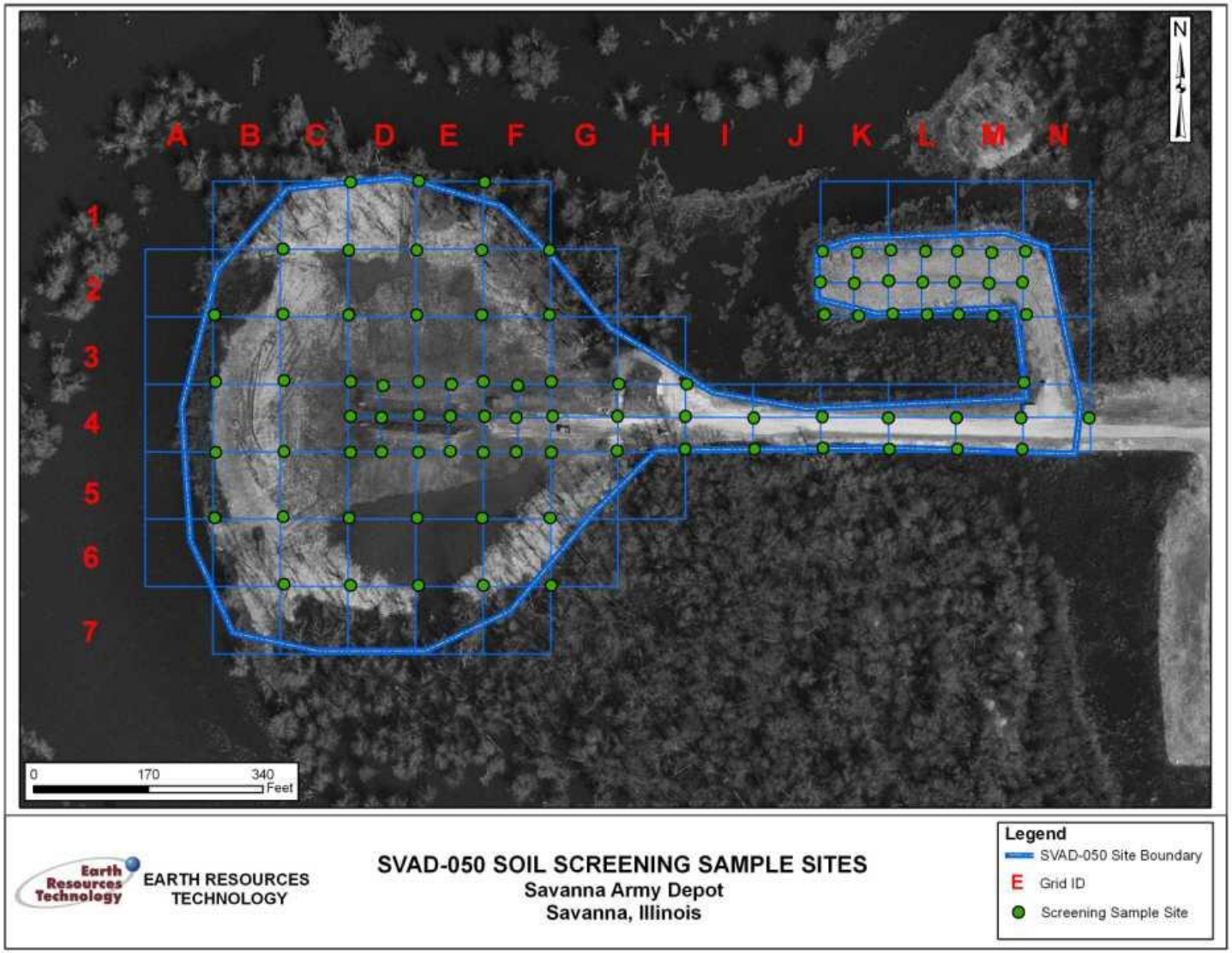


Figure 4. SVAD-050 Soil Screening Sample Sites

Figure 5. SVAD-050 Well Locations

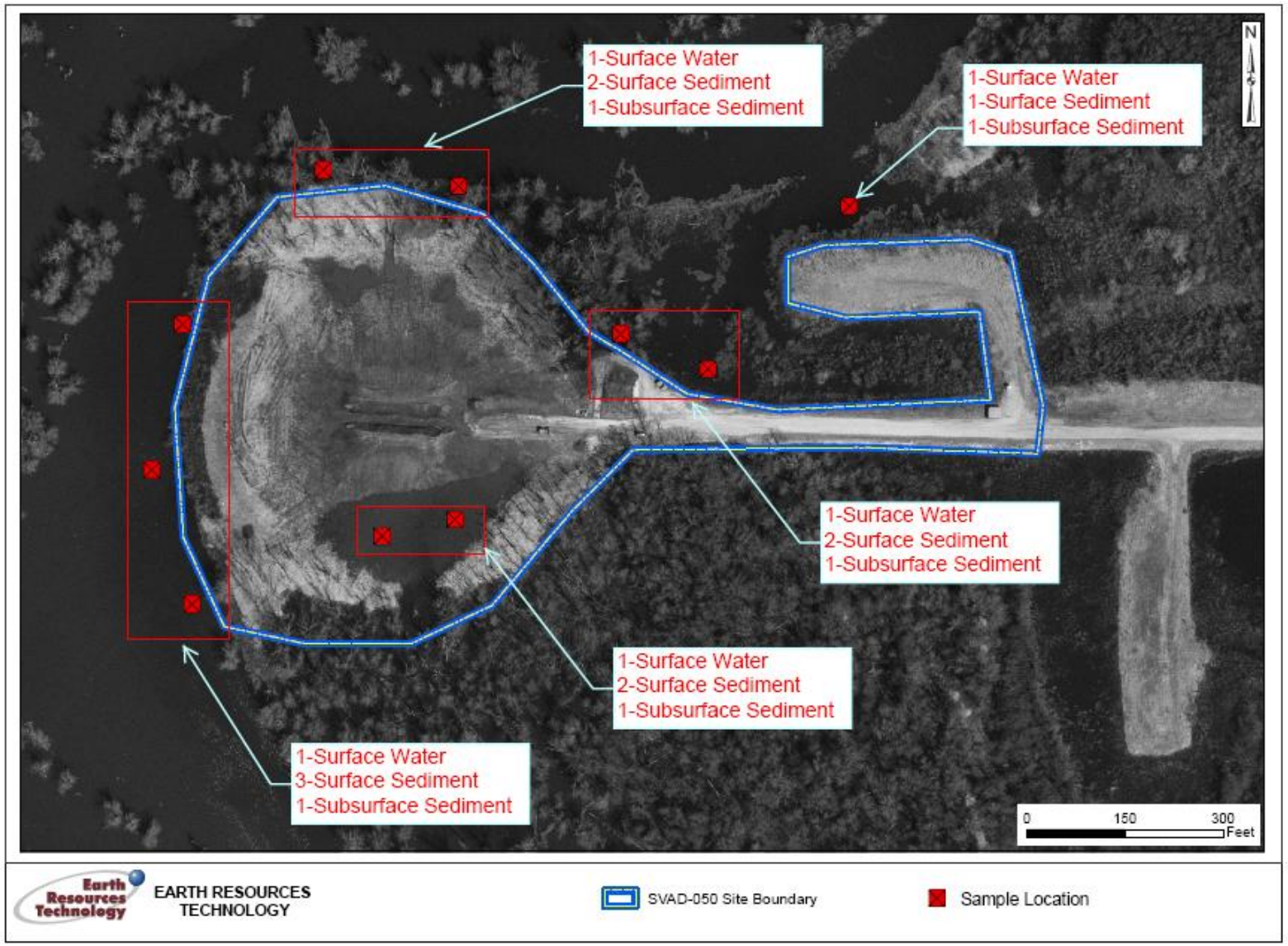


Figure 6. SVAD-050 Sediment and Surface Water Sample Locations

1.6 Secondary Data Evaluation

Table 11. Secondary Data Criteria and Limitations (UFP QAPP Worksheet #13)

Secondary Data	Data Source	Data Generator	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
Assessment of toxic and hazardous materials at SVDA and the potential for these substances to migrate from the Installation.	Installation Assessment	U.S. Army Toxic and Hazardous Materials Agency, 1979	Focus on entire Installation, with reports by interviews of burning of mustard gas packaging materials at Burning Grounds. No sampling and analysis.	Information can be used to associate presence of chlorinated sulfide compounds.
Preliminary environmental sampling and analysis at the OBG.	Rapid Response Environmental Surveys	Environmental Science & Engineering, 1982	Limited monitoring included collection and analysis of 5 soil samples and 18 sediment/surface water samples from a broad area around the site and installing and sampling a single monitoring well.	Data can be used for comparison to RI/FS sampling results.
OBG identified as a solid waste management unit (SWMU).	Groundwater Contamination Survey	U.S. Army Environmental Hygiene Agency, 1988	No sampling and analysis.	No use.
Collection of one confirmatory groundwater sample from the existing monitoring well and three additional surface water samples.	Environmental Monitoring Report	Hunter/ESE, 1989	Limited sample collection. Data confirmed results from 1982 analysis.	Data can be used for comparison to RI/FS sampling results.
Review of historical site monitoring data.	OBG RI/FS	Dames & Moore, 1998	Review of 1982 and 1989 historical sampling data and collection of additional soil and groundwater data. Some analytes had high detection limits.	Data results summarized in SAIC, 2004. SAIC (2002) re-sampled a number of wells.
Evaluation of potential operational problems associated with soil excavation and sifting as a remediation alternative to remove ordnance-related debris.	Ordnance/Explosives Environmental Services Demonstration Project	USACE, 1996	17,300 cubic yards (yd^3) of soil and debris from the eastern portion of the OBG and 3,000 yd^3 from the southern portion successfully excavated, sifted, and stockpiled with pre- and post-excavation soil samples collected and analyzed. TCLP lead criteria not exceeded.	Data can be used for comparison to RI/FS sampling results.

Table 11. Secondary Data Criteria and Limitations (UFP QAPP Worksheet #13)

Secondary Data	Data Source	Data Generator	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
Confirmation sampling and analysis to assess the potential requirements for additional soil remediation.	Soil Retention Area Confirmation Sampling	USACE, Louisville District, 1996	Excavated soil stockpiled included approximately 20,000 yd^3 of soil in five piles. USACE conducted confirmation sampling and analysis to assess the potential requirements for additional soil remediation. The USACE sampling determined that one pile (6,300 yd^3) was characteristically hazardous for lead and the remaining four piles (13,700 yd^3) were nonhazardous.	Data can be used for comparison to RI/FS sampling results.
Sediment, surface water, and soil sampling and soil/sediment toxicity testing.	Supplemental Sediment and Surface Water Investigation	Montgomery Watson, 1998	Investigation relied on data analyses obtained during field activities conducted in 1992 and 1993. The results of the 1997 investigation were inconclusive because of the lack of an adequate background data set for the site and analytical detection limits that exceeded ecological risk assessment guidance values. Supplemental sediment, surface water, and soil sampling and soil/sediment toxicity testing was conducted in October 1997.	Data can be used for comparison to RI/FS sampling results.
Onsite stabilization of characteristically hazardous soils and offsite disposal of the treated material as a special waste.	Stockpile Removal Action	Montgomery Watson, 2000	Data not available.	No use.

Table 11. Secondary Data Criteria and Limitations (UFP QAPP Worksheet #13)

Secondary Data	Data Source	Data Generator	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
Assessment of contaminant toxicity and bioaccumulation that leads to adverse health effects at the OBG due to Army-related contamination in water, sediment, soil, and biota.	Ecological Risk Assessment	Montgomery Watson Harza, 2003	The ERA examined native fish populations, populations of burrowing animals and wetland-dependent vertebrates, plant and invertebrate communities, and state or federally listed threatened and/or endangered species. The field investigation for the ERA included characterization of habitat, collection of samples to characterize levels of chemical exposure, and collection of samples to characterize levels of toxicity. Surface soil, sediment, surface water, and ecological samples (fish tissue, plant, and flying insect) were collected. In addition, MWH conducted background sampling for fish, surface soil, sediment, and surface water.	Data can be used for comparison to RI/FS sampling results.
Groundwater monitoring program at the OBG established and a structural baseline (e.g., visually and physically inspect) and chemical baseline (i.e., sample) of groundwater monitoring wells developed.	Background and Groundwater Sampling	SAIC, 2002	Eleven of the 13 wells sampled by Dames & Moore were redeveloped and three rounds of sampling were conducted. In addition, from 1997 through 2001, SAIC collected background samples for soil, sediment, groundwater, and surface water to enhance the existing background data sets.	Data will be used for comparison to RI/FS sampling results.

Table 11. Secondary Data Criteria and Limitations (UFP QAPP Worksheet #13)

Secondary Data	Data Source	Data Generator	Data Quality Issues	How Data Will Be Used/Limitations on Data Use
Evaluation of risks to receptors under both current and future land uses.	Human Health Risk Assessment	SAIC, 2002	Because of analytical method detection limit issues associated with the older Dames & Moore data, one assessment included data collected by Dames & Moore, MWH, and SAIC, and a second assessment excluded the Dames & Moore data and used only the MWH and SAIC data.	Data can be used for comparison to RI/FS sampling results.
RCRA groundwater monitoring at SVAD-050 and the OBG including sampling for perchlorate in OBG monitoring wells.	Groundwater Sampling	USACE, 2003	Groundwater samples were collected and analyzed from wells 301303 through 301309, 301311, 301402, 301404, and 301405 at the OBG, and wells MW01 to MW10 associated with SVAD-050	Data can be used for comparison to RI/FS sampling results.
RI report prepared.	Old Burning Ground Remedial Investigation Update	SAIC, 2004	Update the report originally prepared by Dames & Moore in 1998 to include additional sampling conducted after 1996 with revised human health and ecological risk assessments.	Data can be used for comparison to RI/FS sampling results.
RCRA groundwater monitoring	RCRA Groundwater Sampling	ERT, 2008-present	Continuation of USACE 2003 efforts.	Data will be used for comparison to RI/FS sampling results.

1.7 Project Overview and Schedule

1.7.1 Project Overview

Table 12. Sampling Tasks (UFP QAPP Worksheet #14)

Soil

Create grid and screen soil samples at node points for explosives.
Identify anomolies and dig test pits.
Collect Phase I samples.
Collect Phase II samples.

Groundwater

Install, develop, and sample new groundwater monitoring well (MW-11).
Sample existing groundwater monitoring wells (RCRA wells).
Collect groundwater levels from top of casing and ground surface using water level indicators.
Translate groundwater levels to elevations using survey or map data.

Sediment

Collect sediment samples.

Surface Water

Collect surface water samples.

Table 13. Analysis Tasks (UFP QAPP Worksheet #14)

CT Laboratories to prepare, process, and analyze TCL VOCs, TCL SVOCs, TAL metals, pesticides, polychlorinated biphenyls (PCB), cyanide, anions, perchlorate, and explosives.

CT Laboratories to analyze Investigative Derived Waste (IDW) from, monitoring well installation and field decontamination procedures by Toxicity Characteristic Leaching Procedure (TCLP).

See Attachment 1, Reference Limits and Evaluation Table, for analytical summaries of soil/sediment, groundwater/surface water, and investigative derived waste (IDW), respectively.

Table 14. Quality Control Tasks (UFP QAPP Worksheet #14)

All matrices will have the following field QC samples analyzed: duplicates, matrix spikes, and matrix spike duplicates. Rinsate blanks and equipment blanks will also be collected for matrices other than groundwater. See Tables 10 and 22 for field QC samples.

Table 15. Data Management Tasks (UFP QAPP Worksheet #14)

All data will be maintained by ERT at ERT Headquarters for a period of at least 5 years. Before disposal, all records will be offered to Mr. Thomas Barounis of USEPA Region 5.

Table 16. Documentation and Records (UFP QAPP Worksheet #14)

A field notebook and forms will be used to record information about each sample along with all field measurements. Each sample will be tracked using chain-of-custody protocol until receipt at the laboratory and using laboratory sample logs afterward. Airbills for overnight courier service will be retained.

ERT will prepare and submit weekly status reports in accordance with Data Item Description (DID) MR-085.

Monthly project status reports that provide the status for the previous month's activities will be submitted to the USACE by the 15th of each month. The monthly report will contain summarized cost and performance information and statistical exposure data for program management purposes. The status reports will include the following information:

- Milestones Achieved (including deliverables submitted)
- Identify if any modifications to Delivery Orders were received
- Task order costs
- Identify project issues or risks
- Upcoming Activities/Milestones
- Identify if any key meetings were held, field work performed, etc.
- Percent complete for each task

Table 17. Assessment/Audit Tasks (UFP QAPP Worksheet #14)

Field Sample Collection and Documentation Audits and Laboratory Technical Systems Audits will be conducted as identified in Table 27, Planned Project Assessments.

Table 18. Data Review Tasks (UFP QAPP Worksheet #14)

The analytical data package will be verified for completeness by the ERT Project Chemist using the project QAPP. Data review checklists will be prepared.

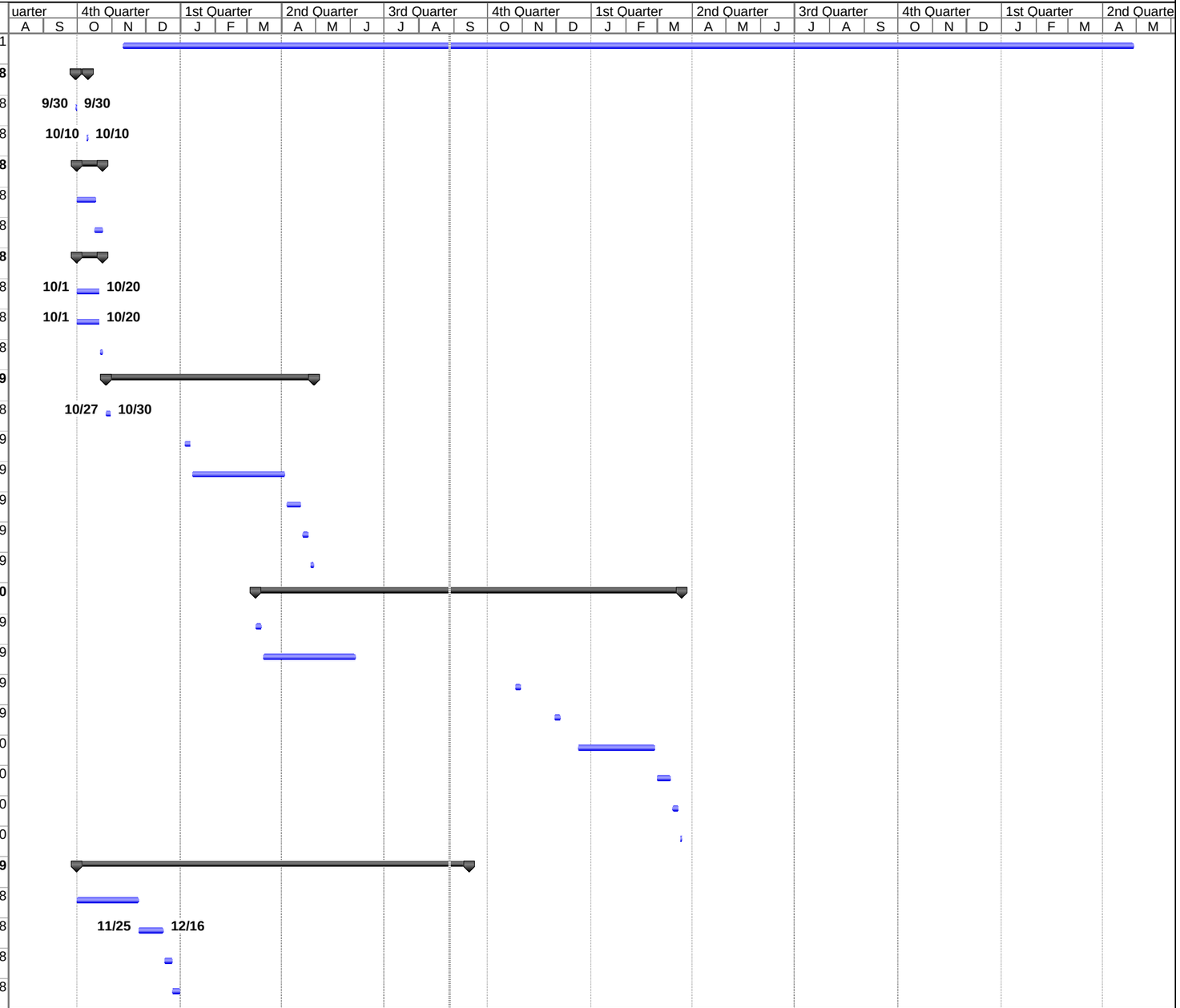
The analytical data package will be validated by an independent third-party reviewer (EDS) using the project QAPP and the appropriate revision of the *National Functional Guidelines for Evaluating Environmental Analysis*. A Tier III Data Validation Report will be prepared for each Sample Delivery Group (SDG) as assigned by the laboratory.

1.7.2 Project Schedule

All project activities that will be performed during the course of the project are included in Figure 7.

Savanna Army Depot, Savanna, IL

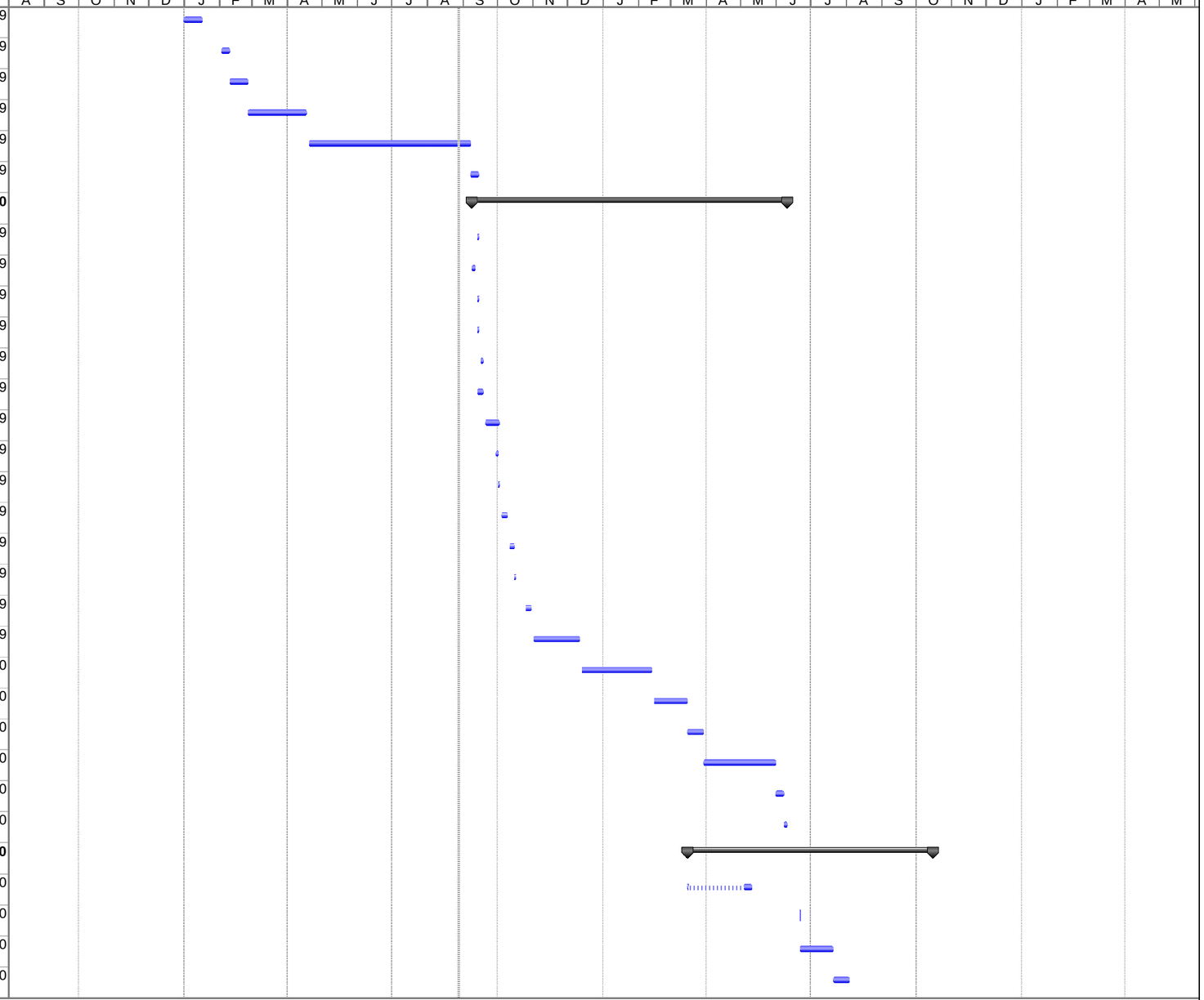
ID	WBS	Task Name	Duration	Start	Finish
1	1	**Savanna Army Depot, IL**	643 days	Tue 11/11/08	Thu 4/28/11
2	**2**	**Award Task Order/ NTP**	**9 days**	**Tue 9/30/08**	**Fri 10/10/08**
3	2.1	Award/ NTP Task Order	1 day	Tue 9/30/08	Tue 9/30/08
4	2.2	Project Kick Off Meeting	1 day	Fri 10/10/08	Fri 10/10/08
5	**3**	**Quality Control Plan (QCP)**	**17 days**	**Wed 10/1/08**	**Thu 10/23/08**
6	3.1	Draft QC Plan	13 days	Wed 10/1/08	Fri 10/17/08
7	3.2	Final QCP	5 days	Fri 10/17/08	Thu 10/23/08
8	**4**	**RCRA Quarterly Sampling**	**17 days**	**Wed 10/1/08**	**Thu 10/23/08**
9	4.1	Sampling & Analysis Plan	14 days	Wed 10/1/08	Mon 10/20/08
10	4.2	Health & Safety	14 days	Wed 10/1/08	Mon 10/20/08
11	4.3	Approve Quarterly Sampling Plan	2 days	Wed 10/22/08	Thu 10/23/08
12	**5**	**2008 RCRA Quarterly Sampling**	**133 days**	**Mon 10/27/08**	**Wed 4/29/09**
13	5.1	Oct 08 Sampling	4 days	Mon 10/27/08	Thu 10/30/08
14	5.2	Dec 08 Sampling	5 days	Mon 1/5/09	Fri 1/9/09
15	5.3	Draft 2008 Sampling Report	60 days	Mon 1/12/09	Fri 4/3/09
16	5.4	Review 2008 Sampling Report	10 days	Mon 4/6/09	Fri 4/17/09
17	5.5	Final 2008 Sampling Report	5 days	Mon 4/20/09	Fri 4/24/09
18	5.6	Approve 2008 Sampling Report	3 days	Mon 4/27/09	Wed 4/29/09
19	**6**	**2009 RCRA Quarterly Sampling**	**271 days**	**Mon 3/9/09**	**Mon 3/22/10**
20	6.1	March 2009 Sampling Round	5 days	Mon 3/9/09	Fri 3/13/09
21	6.2	June 2009 Sampling Round	60 days	Mon 3/16/09	Fri 6/5/09
22	6.3	September 2009 Sampling Round	5 days	Mon 10/26/09	Fri 10/30/09
23	6.4	December 2009 Sampling Round	5 days	Mon 11/30/09	Fri 12/4/09
24	6.5	Draft 2009 Sampling Report	50 days	Mon 12/21/09	Fri 2/26/10
25	6.6	Review 2009 Sampling Report	10 days	Mon 3/1/10	Fri 3/12/10
26	6.7	Final 2009 Sampling Report	5 days	Mon 3/15/10	Fri 3/19/10
27	6.8	Approve 2009 Sampling Report	1 day	Mon 3/22/10	Mon 3/22/10
28	**7**	**RI/FS Work Plans**	**249 days**	**Wed 10/1/08**	**Mon 9/14/09**
29	7.1	Draft RI Workplans	39 days	Wed 10/1/08	Mon 11/24/08
30	7.2	Review Draft RI Workplans	16 days	Tue 11/25/08	Tue 12/16/08
31	7.3	Draft Final RI Workplans	5 days	Thu 12/18/08	Wed 12/24/08
32	7.4	Review Draft Final RI Workplans	5 days	Thu 12/25/08	Wed 12/31/08



Project: Savanna Army Depot
Date: Fri 8/28/09

Task	Baseline Milestone	Baseline Summary	Split	Group By Summary
Progress	Summary	Rolled Up Baseline	Baseline Split	Deadline
Baseline	Rolled Up Task	Rolled Up Baseline Milestone	External Tasks	
Milestone	Rolled Up Milestone	Rolled Up Progress	Project Summary	

ID	WBS	Task Name	Duration	Start	Finish
33	7.5	Revised Draft Final RI/FS Workplans	12 days	Thu 1/1/09	Fri 1/16/09
34	7.6	Review Revised Draft Final RI/FS Workplans	5 days	Tue 2/3/09	Mon 2/9/09
35	7.7	Second Revised Draft Final RI/FS Workplans	12 days	Tue 2/10/09	Wed 2/25/09
36	7.8	Review Second Revised Draft Final RI/FS Workplans	37 days	Thu 2/26/09	Fri 4/17/09
37	7.9	Final RI/FS Workplans	101 days	Mon 4/20/09	Mon 9/7/09
38	7.10	Approve RI/FS Workplans	5 days	Tue 9/8/09	Mon 9/14/09
39	**8**	**RI Field Work**	**197 days**	**Wed 9/9/09**	**Thu 6/10/10**
40	8.1	Kick off Meeting	1 day	Mon 9/14/09	Mon 9/14/09
41	8.11	GPO	3 days	Wed 9/9/09	Fri 9/11/09
42	8.3	Mobilization RI Field Work	1 day	Mon 9/14/09	Mon 9/14/09
43	8.4	Training	1 day	Mon 9/14/09	Mon 9/14/09
44	8.5	Surveying	2 days	Thu 9/17/09	Fri 9/18/09
45	8.6	Clearing	5 days	Mon 9/14/09	Fri 9/18/09
46	8.7	MEC Surface Clearance	10 days	Mon 9/21/09	Fri 10/2/09
47	8.8	DGM Geophysical Mapping	2 days	Wed 9/30/09	Thu 10/1/09
48	8.9	DGM Data Processing	1 day	Fri 10/2/09	Fri 10/2/09
49	8.10	Field Screening Soil Sampling	5 days	Mon 10/5/09	Fri 10/9/09
50	8.11	MEC Investigations	4 days	Mon 10/12/09	Thu 10/15/09
51	8.12	Demobilization	1 day	Fri 10/16/09	Fri 10/16/09
52	8.13	Soil sampling Phase II	5 days	Mon 10/26/09	Fri 10/30/09
53	8.14	Lab Analysis	30 days	Mon 11/2/09	Fri 12/11/09
54	8.15	Draft RI Report	45 days	Mon 12/14/09	Fri 2/12/10
55	8.16	Review Draft RI Report	21 days	Mon 2/15/10	Mon 3/15/10
56	8.17	Draft Final RI Report	10 days	Tue 3/16/10	Mon 3/29/10
57	8.18	Review Draft Final RI Report	45 days	Tue 3/30/10	Mon 5/31/10
58	8.19	Final RI Report	5 days	Tue 6/1/10	Mon 6/7/10
59	8.20	Approve RI Report	3 days	Tue 6/8/10	Thu 6/10/10
60	**9**	**Feasibility Study**	**154 days**	**Tue 3/16/10**	**Fri 10/15/10**
61	9.1	FS Kick off Meeting	6 days	Tue 3/16/10	Mon 5/10/10
62	9.2	Draft FS Report	30 days	Tue 5/11/10	Mon 6/21/10
63	9.3	USACE Drart Report Review	21 days	Tue 6/22/10	Tue 7/20/10
64	9.4	Draft Final FS	10 days	Wed 7/21/10	Tue 8/3/10

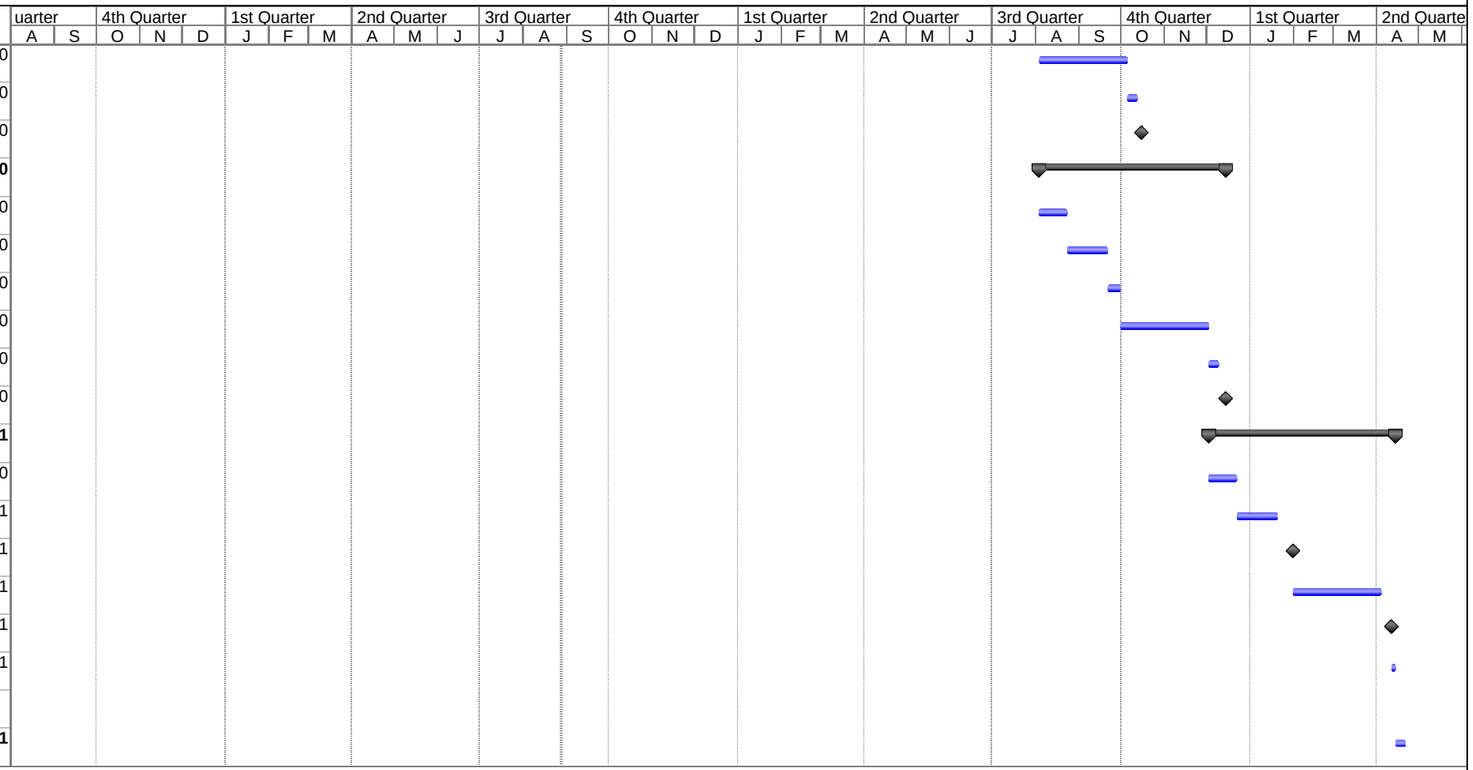


Project: Savanna Army Depot
Date: Fri 8/28/09

Task	Baseline Milestone	Baseline Summary	Split	Group By Summary
Progress	Summary	Rolled Up Baseline	Baseline Split	Deadline
Baseline	Rolled Up Task	Rolled Up Baseline Milestone	External Tasks	
Milestone	Rolled Up Milestone	Rolled Up Progress	Project Summary	

Savanna Army Depot, Savanna, IL

ID	WBS	Task Name	Duration	Start	Finish
65	9.5	Stakeholder Draft Report Review	45 days	Wed 8/4/10	Tue 10/5/10
66	9.6	Final FS	5 days	Wed 10/6/10	Tue 10/12/10
67	9.7	Approve FS	3 days	Wed 10/13/10	Fri 10/15/10
68	**10**	**Proposed Plan (PP)**	**95 days**	**Wed 8/4/10**	**Tue 12/14/10**
69	10.1	Draft PP	14 days	Wed 8/4/10	Mon 8/23/10
70	10.2	USACE Review	21 days	Tue 8/24/10	Tue 9/21/10
71	10.3	Draft Final PP	7 days	Wed 9/22/10	Thu 9/30/10
72	10.4	Stakeholder Draft Review	45 days	Fri 10/1/10	Thu 12/2/10
73	10.5	Final PP	5 days	Fri 12/3/10	Thu 12/9/10
74	10.6	Approve PP	3 days	Fri 12/10/10	Tue 12/14/10
75	**11**	**Record of Decision(ROD)**	**95 days**	**Fri 12/3/10**	**Thu 4/14/11**
76	11.1	Draft DD	14 days	Fri 12/3/10	Wed 12/22/10
77	11.2	USACE Review Draft DD	21 days	Thu 12/23/10	Thu 1/20/11
78	11.3	Draft Final DD	7 days	Fri 1/21/11	Mon 1/31/11
79	11.4	Review Draft Final DD	45 days	Tue 2/1/11	Mon 4/4/11
80	11.5	Final DD	5 days	Tue 4/5/11	Mon 4/11/11
81	11.6	Approve DD	3 days	Tue 4/12/11	Thu 4/14/11
82					
83	**12**	**Project Complete**	**5 days**	**Fri 4/15/11**	**Thu 4/21/11**



Project: Savanna Army Depot
Date: Fri 8/28/09

Task	Baseline Milestone	Baseline Summary	Split
Progress	Summary	Rolled Up Baseline	Baseline Split
Baseline	Rolled Up Task	Rolled Up Baseline Milestone	External Tasks
Milestone	Rolled Up Milestone	Rolled Up Progress	Project Summary

Group By Summary	
Deadline	

2.0 MEASUREMENT DATA ACQUISTION

2.1 Sampling Tasks

2.1.1 Sampling Process Design and Rationale

MEC Survey

Prior to sampling events, a MEC survey and clearance will be conducted for areas of proposed DGM surveys and environmental sampling. The MEC Investigation Plan details activities and safety hazards specific to the task and is presented as Appendix E to the RI/FS Work Plan.

Phase I and II Sampling

It is the intent of these proposed sampling locations to characterize and delineate potential impacts inherent to SVAD-050. After a review of the geophysical investigations and field screening soil sample results (Phase I soil sample results), a second phase of surface and subsurface soil sampling will be conducted. Phase II soil sampling will consist of surface and subsurface samples collected for laboratory analysis from at least 25 locations. The Phase II sampling will also include sampling of groundwater from the new monitoring well (described below), surface water, and surface and subsurface sediment.

DropEx® Field Screen

A systematic sampling approach has been designed to field screen soil samples, in the entire SVAD-050 area (see Figure 4). Sample locations will be located on 50 ft or 100 ft centers, with the exception of where sample locations encounter former structures that are unable to be penetrated. Systematic samples originally placed in such described locations will be offset a minimal distance to remove the obstruction from the sample location. A total of 88 systematic field screening sample locations, based on grid nodes, and 17 additional biased field screening samples will be collected based on field observations during Phase I. These grab surface soil samples will be analyzed for explosives, using DropEx® explosives detection kits.

Geophysical Investigation and Test Pit Excavation Sampling

Geophysical investigations will be conducted across various sections of SVAD-050 prior to sampling activities. The GIP details activity associated with this task and is presented as Appendix D to the Work Plan. Based upon the analysis of the geophysical investigation, six test pit excavations will be executed. As described in the GIP, criteria for selecting test pit excavation locations will be based on factors such as high concentrations of geophysical anomalies. A minimum of one test pit will be excavated at each of the OD and OB areas. Within each excavated test pit, a minimum of two soil samples, surface and subsurface, will be collected immediately adjacent to the anomaly for laboratory analysis of all parameters listed in the Soil Reference Limits and Evaluations Table (Attachment 1).

Phase II Soil Sampling

Two soil samples will be collected from each Phase II sample location for laboratory analysis. One sample, a surface soil sample, will be collected from 0-6 inches below ground surface (bgs). The second soil sample will be collected from the subsurface interval most likely impacted. This interval will be selected based upon field observations, field PID measurements, and information obtained during the geophysical investigation. Each phase II soil sample collected will be

laboratory analyzed for parameters listed in the Soil Reference Limits and Evaluations Table (Attachment 1). Note, if certain analyte groups (such as PCBs or pesticides) are not detected during the Phase I sampling, or if their levels are below screening values and are not considered COPCs, those analytes may be dropped from the Phase II analytical suite.

Groundwater Sampling

One groundwater well will be installed and sampled in the center of the berms of the OD area (see Figure 5). The results of this groundwater sample will be assessed with groundwater data generated from quarterly sampling events at eight wells within SVAD-050, to evaluate potential impacts to groundwater. The groundwater sample will be laboratory analyzed for all parameters listed in the Aqueous Reference Limits and Evaluations Table (Attachment 1).

Surface Water Sampling

Five surface water samples will be collected; one from within the pond located in the OD area and four from the open waterway bordering SVAD-050. The proposed sample locations are presented in Figure 6. The exact location of these samples is unknown and will be predicated upon site condition during field activities. Seasonal flooding occurs at the site and greatly impacts potential sampling locations. At a minimum, surface water samples will be collected from within standing water greater than 12 inches in depth. The surface water samples will be laboratory analyzed for all parameters (except for perchlorate) listed in the Aqueous Reference Limits and Evaluations Table (Attachment 1).

Sediment Sampling

Ten surface (0-6" bgs) and five subsurface (2-3' bgs) sediment samples will be collected; two surface sediment samples and one subsurface sediment sample will be collected from within the pond located in the OD Area, three surface sediment samples and one subsurface sediment sample will be collected in the open waterway to the west of the OD Area, two surface sediment samples and one subsurface sediment sample will be collected in the open waterway to the north of the OD Area; two surface sediment samples and one subsurface sediment sample will be collected in the open waterway between the OD and OB Areas; and one surface sediment sample and one subsurface sediment sample will be collected in the open waterway to the north of the OB Area. The proposed sample locations are presented in Figure 6. The exact location of these samples is unknown and will be predicated upon site condition during field activities. Seasonal flooding occurs at the site and greatly impacts potential sampling locations. At a minimum, sediment samples will be collected from within standing water greater than 12 inches in depth. For the purpose of the RI, sediment will be considered soils present below the mean water level of any static or open water body that is saturated greater than 50% of the year. This definition is intended to exclude soils which lie at the base of puddles, drainage ditches, swales and other similar formations. Mean water level will be established by visual observation of shoreline water marks and/or by interviews with on-site personnel. The sediment samples will be laboratory analyzed for all parameters listed in the Soil Reference Limits and Evaluations Table (Attachment 1).

Schedule and Considerations

Field work for the SVAD-050 RI/FS will be conducted during the Fall of 2009 during baseline flow conditions (i.e. this is a non-runoff related event).

Table 19. Sampling Locations and Methods (UFP QAPP Worksheet #18)

Sampling Identity	Matrix	Depth	Analytical Groups	Detection Level[1]	Number of Samples[2]	Rationale for Sampling Location
Phase I Soil Entire SVAD-050 Area	Soil	Surface 0-6" bgs	Explosives	Field Screening	116	Systematic approach to screen entire area.
Phase I Soil Test Pits (6)	Soil	Surface 0-6"bgs	TCL VOCs TCL SVOCs TAL metals Pesticides PCBs Cyanide Explosives	Low Level	6	Based upon geophysical investigation.
Phase I Soil Test Pits (6)	Soil	Subsurface 1-15' bgs		Low Level	7	Based upon geophysical investigation , PID measurements, and observations.
Phase II Soil	Soil	Surface 0-6"bgs		Low Level	27	Based upon Field Screen, observations, and information obtained during the geophysical investigation.
Phase II Soil	Soil	Subsurface 1-15' bgs		Low Level	28	Based upon PID measurements, observations, and information obtained during the geophysical investigation.
Phase II Groundwater	Groundwater[3]	TBD	TCL VOCs TCL SVOCs TAL metals Pesticides PCBs Cyanide Explosives Anions Perchlorate (GW only)	Low Level	2	Well to be installed and sampled in center of OD berms (center of historical OD activity)
Phase II Pond	Surface Water	>12" water		Low Level	2	Pond is within SVAD-050 boundaries and subject to contamination.
Phase II Waterways	Surface Water	>12" water		Low Level	4	Waterways form border of SVAD-050.
Phase II Pond	Sediment	Surface 0-6" bgs Within water having > 12" depth	TCL VOCs TCL SVOCs TAL metals Pesticides PCBs Cyanide Explosives	Low Level	3	Pond is within SVAD-050 boundaries and subject to contamination.

Table 19. Sampling Locations and Methods (UFP QAPP Worksheet #18)

Sampling Identity	Matrix	Depth	Analytical Groups	Detection Level[1]	Number of Samples[2]	Rationale for Sampling Location
Phase II Pond	Sediment	Subsurface 2-3' bgs Within water having > 12" depth	TCL SVOCs TAL metals Pesticides PCBs Cyanide Explosives	Low-Level	2	Pond is within SVAD-050 boundaries and subject to contamination.
Phase II Waterways	Sediment	Surface 0-6" bgs Within water having > 12" depth		Low Level	8	Waterways form border of SVAD-050.
Phase II Waterways	Sediment	Subsurface 2-3' bgs Within water having > 12" depth		Low-Level	4	Waterways form border of SVAD-050.

[1] See Attachment 1 for Laboratory Method Detection Limits (MDLs)
[2] Includes duplicate samples
[3] All groundwater and associated QC samples from MW-11 will be sampled in conjunction with the fourth quarter 2009 RCRA sampling at the OB/OD area

2.1.2 Sampling Procedures and Requirements

ERT's SOPs for the RI/FS field activities associated with environmental sampling at SVAD-050 are presented in Attachment 3, and corresponds to the SOP reference table (Table 20), below.

Table 20. ERT's Standard Operating Procedures (UFP QAPP Worksheet #21)

Category	SOP Title	Date, Revision	SOP #
Water-related Field Activities	Monitoring Well Installation	01/22/09, Rev 1	05-2
	Low-flow Groundwater Sampling	01/22/09, Rev 1	06-5
	Well Development	01/22/09, Rev 1	05-4
	Well Purging – Pumping Method	01/22/09, Rev 1	05-6
	Water Level Measurements	01/22/09, Rev 1	07-2
	Surface Water and Sediment Sampling	01/22/09, Rev 1	06-2
	Field Filtration of Water Samples	01/22/09, Rev 1	06-8
Soil-related Field Activities	Borehole Logging	01/22/09, Rev 1	07-3
	Soil Sampling	01/22/09, Rev 1	06-3
	PID Operation	03/24/09, Rev 1	06-13
	Explosives Field Screening	03/25/09, Rev 1	06-10
Waste-related Field Activities	IDW Management	01/22/09, Rev 1	06-4
	Explosives Field Screening	03/25/09, Rev 1	06-10
	Drum Handling and Sampling	03/24/09, Rev 1	06-14
Decontamination Procedures	Decontamination of Well Installation Equipment and Materials	01/22/09, Rev 1	03-2
	Decontamination of Sampling Equipment	01/22/09, Rev 1	03-1
Preparation/Completion Activities	Preparing for and Concluding Field Activities	01/22/09, Rev 1	01-1
	Monitoring Well and Borehole Abandonment	01/22/09, Rev 1	05-7
Documentation and Custody Procedures	Use of Field Logbooks	01/22/09, Rev 1	01-2
	Packaging and Shipment of Field Samples	01/22/09, Rev 1	06-7

2.1.2.1 Sampling Collection Procedures

The following sections summarize the procedures to be used during the field sampling efforts.

Explosives Field Screening

Field screening to provide a rapid environmental screen of surface soil samples collected along the path of the historic TNT Lines in SVAD-050 will be conducted using DropEx® Explosion Detection/Identification Field Test Kits. DropEx® is an liquid–based detection system for the

identification of Group A and B explosives through a series of sequential reactions. Nitroaromatics, nitrate esters and nitramines are determined by a series of reactions which differentiates between the concentrations and sets of compounds within the media tested.

Soil Sampling (Hand Auger)

Soil sampling will be performed by advancing a hand auger to the desired depth below the surface. All subsurface intervals (in the unsaturated soil extending from the ground surface to the water table) will be visually inspected for staining, discoloration, odors, and debris indicative of contamination, and PID measurements will be run over the length of the soil boring to aid in determining the appropriate grab sampling interval. PID readings will be used to as a secondary indicator to assist in determining sampling interval when other obvious characteristics (such as staining, discoloration, or odor) are not present."A small digging instrument will be used to break or expose fresh soil (from hand augured soil borings) that hasn't been exposed to the atmosphere. The tip of the PID will be placed in very close proximity to the freshly exposed soil and a measurement taken.

All samples will be collected by cutting the soil in two places with a decontaminated steel, stainless steel, or aluminum trowel, spoon or knife and placed in the sample bottles. Samplers will wear phthalate-free gloves such as nitrile (no latex will be used) and will avoid contact of the gloves with the sample. Only clean metal instruments will be allowed to touch the sample.

Field conditions may require a section longer than six inches to be used in order to obtain sufficient soil volume for sample analysis. This decision will be field-based, and if it occurs, then the sample volume from the insufficient section will be composited with adjacent sections.

Hand auguring will be the preferred method for obtaining subsurface soil samples; however, other drilling methods including disposable scoops/troughs, mud rotary and drive and wash may also be used if warranted by site conditions.

Soil Sampling (Direct Push)

Soil samples may be collected utilizing a direct push, Geoprobe 6620® or similar. Direct push is the preferred method for the installation of the lone groundwater monitoring well to be installed in the OD Area. Organic vapor screening and soil collection from the acetate sleeve in this procedure will follow the method described above.

Sediment Sampling

Grab sediment samples will be collected from the open waterway adjacent to SVAD-050 to delineate the extent and migration of potential contamination. Surface sediment samples will be collected from 3-6" bgs and grab subsurface sediment samples will be collected from 2-4' bgs in water at least 12" in depth, and also below the mean high water line along the shores of the water body (established by visual observation of shoreline water marks and/or by interviews with on-site personnel).

Samplers will wear phthalate-free gloves such as nitrile (no latex will be used) and will avoid contact of the gloves with the sample. Only clean metal instruments will be allowed to touch the sample. If there is insufficient soil volume, then this will be made up by attempting a second core at the same depth, if appropriate.

The surface sediment samples will be collected by hand auguring a core to the required depth with a decontaminated steel, stainless steel, or aluminum trowel, or disposable scoop or knife and placed in the sample bottles. Subsurface sediment samples will be collected by either hand auger and tube sampler or hand driven split spoon core-sampler and placed in the sample bottles.

Each dedicated bottle will be opened and screened using PID to detect possible organic vapors. The samples will be also examined for staining, discoloration, odors, and debris indicative of contamination.

Surface Water Samples

Surface water samples will be taken starting at the furthest downstream sampling location and then sequentially moving in an upstream direction. Kemmerer (adapted with Teflon stoppers) or equivalent bottle sampler will be utilized to collect a sample, and then the water will be transferred to the appropriate containers.

Groundwater Sampling

Groundwater samples will be collected using low-flow methodology (e.g. bladder pump) to minimize loss of volatiles and turbidity in the samples.

Groundwater parameters (pH, temperature, conductivity, ORP, turbidity, and DO) will be monitored with a Horiba U-22 Multiparameter Water Quality Monitoring System, and water level will be monitored with an electric water level meter. Once groundwater conditions have stabilized, all samples will be collected from the Teflon-lined polyethylene tubing at the flow cell outlet.

Sampling will be performed by suspending the bottom of the pump a minimum of 15 inches above the bottom of the well and a minimum of 0.5 feet from the top of the water level, in order to minimize the amount of suspended sediment in the sample. Pumping rates for withdrawing the samples will be similar to those followed for well purging.

Liquid Waste Characterization

Investigative derived waste from equipment decontamination procedures and purge/development water from the newly installed monitoring well (MW-11) will be containerized on-site in 55-gallon drums and transported by a subcontractor for proper off-site disposal. Environmental (GW, SW, soil, sediment) sample results will be sent to the waste disposal facility in advance of the IDW for determination of proper disposal method. Upon request of the disposal facility, the liquid IDW may also be sampled and characterized further for TCLP analyses and RCRA characteristics (see IDW Reference and Limits Table, Attachment 1). If other analyses become necessary, the parameters and methods will be provided as an addendum to this QAPP.

If deemed necessary, liquid IDW sampling methods will utilize dedicated dippers and glass tube samplers. Dippers are used to collect samples from the surface of the liquid, and are appropriate for wastes that are homogeneous. Glass tube samplers consist of glass tubes of varying length and diameter used to collect a full-depth liquid sample from a drum or similar container.

Solid Waste Characterization

All scrap munitions debris (MD) uncovered during the MEC Survey will be collected, screened for residual explosives with Expray® or DropEx®, and containerized in 55-gallon drums. If other analyses become necessary, the parameters and methods will be provided as an addendum to this QAPP. Scrap that tests negative for explosives will be handled and disposed of as non-hazardous waste and scrap that tests positive for explosives will be handled and disposed of seperately.

Soil cuttings recovered during the installation of MW-11 will be containerized in a 55-gallon drum. Waste soil will be sampled by advancing a hand auger through the containerized volume of soil (not just grabbing a sample off the top) and characterized for TCLP analyses and RCRA characteristics (see IDW Reference and Limits Table, Attachment 1). If other analyses become necessary, the parameters and methods will be provided as an addendum to this QAPP.

The containerized MD wastes and any soil determined to be hazardous will be transported by a subcontractor to the proper off-site disposal facility(s).

Grab/Composite Sampling

Grab sampling is the preferred method for all matrices, though compositing may be necessary for soil cutting wastes. If it is deemed necessary to characterize the liquid or solid IDW by TCLP methods and more than 1 drum is used, then samples from the drums will be composited for TCLP analyses.

2.1.2.2 Sample Containers, Volume, and Preservation

For all matrices, samples for VOC analysis will be obtained first. All aqueous VOC sample bottles must be filled to the top so that no head space or air bubbles will be left in the sample containers, thereby reducing aeration of the samples during transport and minimizing any precipitation of colloidal matter. When collecting aqueous VOC samples, the water should be allowed to run down the side of the collection bottle. For samples not being analyzed for VOCs, an adequate headspace will be left to compensate for differences in barometric pressure encountered during sample shipment. Aqueous samples for dissolved metals will be collected in unpreserved containers and will be filtered and preserved at the laboratory within 24 hours of sampling.

The analytical laboratory will supply the sample containers for the chemical samples. These containers will be cleaned by the manufacturer to meet or exceed all analyte specifications established in the latest EPA's *Specifications and Guidance for Contaminant-Free Sample Containers (EPA, 1992)*. Certificates of analysis are provided with each bottle lot and maintained on file to document conformance to EPA specifications. Volumes and preservation methods will comply with respective method requirements.

Upon collection, samples will be labeled with a unique alpha-numerical field sample number based on the established labeling system detailed in the USACE SOW dated September 29, 2008. Samples will be immediately wrapped in protective bubble wrap and placed on ice for shipment. All samples will be shipped within 24 hours of collection. Samples will be sent to CT Laboratories, Inc. for analysis.

Table 21 presents the sample number, types, and method requirements to be followed in the SVAD-050 RI/FS. Table 22 presents a field QA/QC sample summary.

Table 21. Analytical Parameters, Methods, Preservation and Container Requirements (UFP QAPP Worksheet #19)

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Soil/Sediment	VOCs (TCL)	Grab	77	SW-846 Method 8260B	Cool to 4^0 C; no headspace	14 days to analysis	(1) Terra Core kit (Low-level)
Soil/Sediment	PCBs	Grab	77	SW-846 Method 8082	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(1) 9-oz amber glass jar
Soil/Sediment	Pesticides (TCL)	Grab	77	SW-846 Method 8081A	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	SVOCs (TCL)	Grab	77	SW-846 Method 8270C	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Metals (TAL)	Grab	77	SW-846 Method 6010B/7470A	Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 9-oz glass jar
Soil/Sediment	Cyanide	Grab	77	SW-846 Method 9012A	Cool to 4^0 C	14 days to analysis	
Soil/Sediment	Explosives	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(2) 9-oz glass jars
Soil/Sediment	Nitrocellulose	Grab	77	SW-846 Method 9056M	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroguanidine	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	
Soil/Sediment	Nitroglycerine	Grab	77	SW-846 Method 8330B	Cool to 4^0 C	7 14 days to extraction; 40 days from extraction to analysis	

Table 21. Analytical Parameters, Methods, Preservation and Container Requirements (UFP QAPP Worksheet #19)

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Groundwater/Surface Water	VOCs (TCL)	Grab	6	SW-846 Method 8260B	pH<2 with HCl; Cool to 4^0 C; no headspace	14 days to analysis	(5) 40 mL VOA vials
Groundwater/Surface Water	SVOCs (TCL)	Grab	6	SW-846 Method 8270C	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater/Surface Water	Metals- total (TAL)	Grab	6	SW-846 Method 6010B	pH<2 with HNO_3; Cool to 4^0 C	28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Groundwater/Surface Water	Metals-dissolved (TAL)	Grab	6	SW-846 Method 6010B/7470A	pH<2 with HNO_3; Cool to 4^0 C	24 hours to filtering and preservation (pH<2 with HNO_3); 28 days to analysis for Hg; 6 months to analysis for other metals	(1) 1L polyethylene bottle
Groundwater/Surface Water	Total Cyanide	Grab	6	SW-846 Method 9012A	pH>12 with NaOH; Cool to 4ºC	14 days to analysis	(1) 500 mL polyethylene container
Groundwater/Surface Water	Nitrate+Nitrite	Grab	6	SW-846 Method 9056	pH<2 with H_2SO_4; Cool to 4^0 C	28 days to analysis	(1) 125 mL polyethylene bottle
Groundwater/Surface Water	Pesticides (TCL)	Grab	6	SW-846 Method 8081A	Cool to 4^0 C	7 days to extraction; 40 days from extraction to analysis	(2) 1L amber glass bottle
Groundwater/Surface Water	PCBs	Grab	TBD	SW-846 Method 8082	Cool to 4^0 C	14 days to extraction; 40 days from extraction to analysis	(1) 1L amber glass jar
Groundwater/Surface Water	Explosives	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	(2) 1L amber glass bottle

Table 21. Analytical Parameters, Methods, Preservation and Container Requirements (UFP QAPP Worksheet #19)

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Groundwater/Surface Water	Nitroguanidine	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Groundwater/Surface Water	Nitroglycerine	Grab	6	SW-846 Method 8330B	Cool to 4ºC	7 days to analysis	
Groundwater/Surface Water	Nitrocellulose	Grab	6	SW-846 Method 9056M	Cool to 4ºC	7 days to analysis	
Groundwater	Perchlorate	Grab	1	SW-846 Method 6850	Cool to 4ºC	28 days to analysis	(1) 125 mL polyethylene container
Investigative Derived Waste (IDW) – Soil/Solids	TCLP VOC (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/8260B	Cool to 4^0 C	14 days to TCLP extraction; 14 days from TCLP extraction to analysis	(1) 32-oz glass jar (1) 9-oz glass jar
Investigative Derived Waste (IDW) - Soil/Solids	TCLP SVOC (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/ 8270C	Cool to 4^0 C	14 days to TCLP extraction; 7 days from TCLP extraction to SVOC extraction; 40 days from SVOC extraction to analysis	
Investigative Derived Waste (IDW) - Soil/Solids	TCLP Pesticides (RCRA)	Grab	TBD/ up to 4[6]	SW-846 Methods 1311/8081A	Cool to 4ºC	14 days to TCLP extraction; 7 days from TCLP extraction to pesticide extraction; 40 days from pesticide extraction to analysis	
Investigative Derived Waste (IDW) - Soil/Solids	TCLP Metals (RCRA)	Grab	TBD/ up to 4[6]	SW 846 Methods 1311/ 6010B/7470A	Cool to 4^0 C	Hg: 28 days to TCLP extraction; 28 days from TCLP extraction to analysis Other Metals: 6 months to TCLP extraction; 6 months from TCLP extraction to analysis	

Table 21. Analytical Parameters, Methods, Preservation and Container Requirements (UFP QAPP Worksheet #19)

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Investigative Derived Waste (IDW) - Soil/Solids	Ignitability	Grab	TBD/ up to 4[6]	SW-846 Method 1010/1030	Cool to 4^0 C	None specified	
Investigative Derived Waste (IDW) - Soil/Solids	Corrosivity	Grab	TBD/ up to 4[6]	SW-846 Method 9045C	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) - Soil/Solids	Reactive cyanide	Grab	TBD/ up to 4[6]	SW-846 Chapter 7, Section 7.3.3	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) - Soil/Solids	Reactive sulfide	Grab	TBD/ up to 4[6]	SW-846 Chapter 7, Section 7.3.4	Cool to 4^0 C	Within 3 days of collection	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP VOC (RCRA)	Grab	TBD	SW 846 Methods 1311/8260B	Cool to 4^0 C	14 days to TCLP extraction; 14 days from TCLP extraction to analysis	(3) 1L glass amber bottles (2) 500mL polyethylene bottle (1) 250mL polyethylene bottle
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP SVOC (RCRA)	Grab	TBD	SW 846 Methods 1311/ 8270C	Cool to 4^0 C	14 days to TCLP extraction; 7 days from TCLP extraction to SVOC extraction; 40 days from SVOC extraction to analysis	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP Pesticides (RCRA)	Grab	TBD	SW-846 Methods 1311/8081A	Cool to 4ºC	14 days to TCLP extraction; 7 days from TCLP extraction to pesticide extraction; 40 days from pesticide extraction to analysis	
Investigative Derived Waste (IDW) – Decontamination Liquid	TCLP Metals (RCRA)	Grab	TBD	SW 846 Methods 1311/ 6010B/7470A	Cool to 4^0 C	Hg: 28 days to TCLP extraction; 28 days from TCLP extraction to analysis Other Metals: 6 months to TCLP extraction; 6 months from TCLP extraction to analysis	

Table 21. Analytical Parameters, Methods, Preservation and Container Requirements (UFP QAPP Worksheet #19)

Sample Matrix	Analytical Parameter	Sample Type[1]	No. of Samples[2]	EPA Analytical Method	Sample Preservation	Holding Time[3]	Sample Container[4,5]
Investigative Derived Waste (IDW) – Decontamination Liquid	Ignitability	Grab	TBD	SW-846 Method 1010/1030	Cool to 4^0 C	None specified	
Investigative Derived Waste (IDW) – Decontamination Liquid	Corrosivity	Grab	TBD	SW-846 Method 9045C	Cool to 4^0 C	As soon as possible (within 3 days of collection)	
Investigative Derived Waste (IDW) – Decontamination Liquid	Reactive cyanide	Grab	TBD	SW-846 Chapter 7, Section 7.3.3	Cool to 4^0 C	As soon as possible (within 3 days of collection)	
Investigative Derived Waste (IDW) – Decontamination Liquid	Reactive sulfide	Grab	TBD	SW-846 Chapter 7, Section 7.3.4	Cool to 4^0 C	As soon as possible (within 3 days of collection)	

[1] For soil samples, a six-inch sampling interval is the preferred sample size; however, sample volume recovery, analytical method requirements, and field conditions can affect the actual sample interval size. For these reasons, the actual sampling interval may change in order to obtain adequate volume.
[2] Actual number of samples may vary depending on field conditions, sample material availability, and field observations. **Does not include QA samples**.
[3] From date of sample collection
[4] I-Chem Series 300 bottles
[5] MS/MSDs require duplicate volume for all parameters for solid matrices; MS/MSDs require triplicate volume for organic parameters for aqueous matrices and duplicate volume for inorganic parameters for aqueous matrices
[6] Up to 4 soil samples collected during Phase II may be analyzed using TCLP.
TBD = To Be Determined

Table 22. Summary of Field Quality Control Samples (UFP QAPP Worksheet #20)

Parameters	SW-846 Analytical Method	Number of Regular Samples	Field Duplicate Samples	Trip Blanks	Rinsate Blanks	FB Samples	MS/ MSDs/Lab Duplicates	Total Analyses
SOIL/SEDIMENT								
TCL VOCs	8260B	77	8	15	4	0	4/4	112
TCL SVOCs	8270C	77	8	NA	4	0	4/4	104
TCL PCBs	8082A	77	8	NA	4	0	4/4	104
TCL Pesticides	8081A	77	8	NA	4	0	4/4	104
Explosives	8330B	77	8	NA	4	0	4/4	104
TAL Metals	6010B	77	8	NA	4	0	4/4	104
Total Cyanide	335.3	77	8	NA	4	0	4/4	104
SURFACE WATER/GROUNDWATER[1]								
TCL VOCs	8260B	6	2	2	1	1	1/1	14
TCL SVOCs	8270C	6	2	NA	1	1	1/1	12
TCL PCBs	8082A	6	2	NA	1	1	1/1	12
TCL Pesticides	8081A	6	2	NA	1	1	1/1	12
Explosives	8330B	6	2	NA	1	1	1/1	12
TAL Metals (total)	6010B	6	2	NA	1	1	1/1	12
TAL Metals (diss)	6010B	6	2	NA	1	1	1/1	12
Nitrate	353.2	6	2	NA	1	1	1/1	12
Nitrite	353.2	6	2	NA	1	1	1/1	12
Total Cyanide	335.3	6	2	NA	1	1	1/1	12
Perchlorate	6850	1	1	NA	0	0	1/1	4

[1] All groundwater and associated QC samples from MW-11 will be sampled in conjunction with regular RCRA sampling at the OB/OD Areas

2.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures

The ERT SOP for equipment decontamination was prepared to direct ERT personnel in the methods for decontamination of field equipment used in hazardous waste investigations. The SOP conforms to "*A Compendium of Superfund Field Operations Methods (EPA/540/P-87/001),"* and other pertinent technical publications.

The objective of equipment decontamination is to remove potential contaminants from a sampling device or item of field equipment prior to and between collection of samples for laboratory analysis and limit personnel exposure to residual contamination that may be present on used field equipment.

Decontamination Equipment

The following items may be utilized when decontaminating equipment. Site-specific conditions may warrant the use or deletion of items from this list.

- Alconox, liquinox or other non-phosphate concentrated laboratory grade soap;
- Deionized Water;
- Pump Sprayer;
- 1-Pint Squeeze bottle filled with pesticide-grade hexane;
- 1-Pint Squeeze bottle filled with pesticide-grade methanol;
- 1-Pint Squeeze bottle filled with ten (10) percent nitric acid;
- 1-Pint Squeeze bottle filled with one (1) percent nitric acid;
- Five large plastic wash basins (24 inches by 30 inches by 6 inches deep);
- Five large plastic wash basins (24 inches by 30 inches by 6 inches deep);
- Two coarse scrub brushes;
- Small wire brush;
- Aluminum foil;
- Polyethylene sheeting;
- Two large capacity barrels;
- All necessary personal protective equipment (gloves, eyewear, tyveks);
- Extra quantities of above listed liquids; and
- 4 inch Schedule 40 PVC pipe 4 feet in length with an end cap for deconning pump and associated tubing (if needed).

Decontamination Procedures for Aqueous Monitoring Equipment

It is anticipated that only a water level meter and multiparameter water quality instrument will require decontamination, as dedicated tubing will be used with a peristaltic pump, or a dedicated stainless steel bailer will be used to collect samples. Measuring equipment will be decontaminated utilizing the following procedures.

1) Rinse with potable/tap water
2) Scrub with non-phosphate detergent, such as Alconox®
3) Rinse with distilled water

Decontamination Procedures for Soil, Sediment, and Water Sampling Equipment

Soil sampling equipment (such as split spoon samplers, shovels, augers, trowels, and spoons) and groundwater sampling equipment that is not a dedicated or disposable sampling apparatus will be cleaned using the following procedure.

1) Lay out sufficient polyethylene sheeting on the ground or floor to allow placement of the five plastic wash basins and an air drying area. Place wash basins on the polyethylene sheeting.
2) Fill the first wash basin with potable tap water. Add sufficient soap powder or solution to cause suds to form in the basin. Do not use an excessive amount of the soap or rinsing the soap residue off the equipment will be difficult.
3) Using a clean, coarse scrub brush, wash the sampling equipment in the soap solution in the first basin, removing all dirt. Allow excess soap to drain off the equipment when finished.
4) Rinse the equipment with tap water in the second basin, using a coarse scrub brush or pressure sprayer to aid in the rinse, if necessary.
5) If the equipment is being used to sample for metals, rinse the equipment with nitric acid in the third basin. A 10 percent solution is used on stainless steel equipment. A one percent solution is used on all other equipment. If no metals sampling is being performed, this step may be omitted.
6) Spray down the equipment in the third basin, using deionized water.
7) Spray down the equipment in the fourth basin, using pesticide-grade methanol, if sampling for organic compounds is to be performed. If oily, a two-step process using methanol, followed by hexane should be used to remove both water soluble and non-soluble compounds. If no samples for organic compounds are being collected, this step may be omitted.
8) Allow the equipment to completely air dry on clean polyethylene sheeting.
9) Rinse the equipment in the fifth basin, using deionized water.
10) Allow the equipment to completely air dry on clean polyethylene sheeting.
11) Reassemble equipment, if necessary, and wrap completely in clean, unused aluminum foil, shiny side out for transport. Re-use of equipment on the same day without wrapping in foil is acceptable.

12) All spent cleaning solutions shall be drummed for disposal along with any other contaminated fluids generated during the field investigation (per ERT SOP #06-4, IDW Management).
13) Record the decontamination procedure in the field logbook or on appropriate field form.

If step 8, rinsing with organic solvents, was performed, check the equipment for the presence of residual solvents with a PID or flame ionization detector prior to use. If detection occurs, disassemble the equipment and allow to air dry until no readings are observed, then re-rinse with deionized water.

Note that if temperature or humidity conditions preclude air drying equipment, sufficient spares should be available so that no item of sampling equipment needs be used more than once. Alternatively, the inability to air dry equipment completely prior to reuse should be noted in the field logbook. In this case, additional rinses with deionized water should be performed and recorded.

2.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures

General

Field instruments will be calibrated, operated, and maintained in accordance with manufacturer's instructions and specific instrument SOPs. Daily, and in some cases, more frequently, calibration of equipment will provide QC checks on all equipment used during the performance of project activities. Each instrument will have an individual identification number affixed. This number will be transcribed on field data records when using a particular instrument for a sampling event. All calibration, repair, and service records will be kept in individual equipment logbooks maintained for each instrument. Equipment that consistently falls out of calibration or exceeds manufacturer's critical limits will be repaired or replaced. Table 22 summarizes field equipment calibration, maintenance, testing, and inspection.

Photo Ionization Detector (PID)

PIDs can measure total organic vapors and are highly sensitive to aromatic compounds, moderately sensitive to unsaturated chlorinated compounds, and less sensitive to aliphatic hydrocarbons. The instrument can respond to organic compounds with ionization potentials less than the rated eV of the ultraviolet bulb in the unit. Due to its longevity and range of detectable contaminants, the most frequently used ultraviolet bulb is a 10.2-eV. However, in order to detect the whole suite of VOC's, an 11.2-eV bulb will be used.

Several manufacturers produce PIDs for field monitoring of airborne VOC's. The field technician will follow the manufacturer's calibration requirements. General guidance for PID calibration includes:

- Factory service and calibration once per year
- For any PID instrument, a two-point calibration before daily use (UHP air and a representative concentration of isobutylene in air standard)

Table 23. Field Equipment Calibration, Maintenance, Testing, and Inspection (UFP QAPP Worksheet #22)

Field Equipment	Calibration	Maintenance	Testing	Inspection	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
HORIBA U-22	Auto-cal	Decon between each location. Check batteries.	Field test according to manual	Inspect for external damage	Daily	Within cal standard range	Recalibrate. Replace batteries.	Field Personnel	06-5
Electronic Water Level Indicator	According to manufacturer	Decon between each location. Check batteries.	Field test according to manual	Inspect tape for kinks and cuts. Inspect probe for dirt.	Daily	Response	Replace battery if no response.	Field Personnel	07-2
Photoionization Detector (PID)	According to manufacturer	Check batteries.	Field test according to manual	Inspect probe for dirt. Inspect for external damage.	Daily	Within cal standard range	Recalibrate. Replace batteries	Field Personnel	06-3

2.1.2.5 Supply Inspection and Acceptance Procedures

Respective ERT SOPs presented in Attachment 3 provide the procedures and activities that will be performed in the field to ensure that all sampling supplies are free of target analytes/COPC and interferences. Incorporation of various QA/QC blanks (field, rinsate, and VOC trip blanks) will be employed to measure and ensure supply and handling cleanliness. In addition, all sample collection containers will be provided by the contract laboratory. All analytical supplies and reagents that could affect data quality will be certified and a record of associated lot numbers will be maintained.

2.1.2.6 Field Documentation Procedures

Field activity documentation is one of the most important activities that occur in a field sampling task. There is abundant information available for documenting the details of a sampling effort at the time the sampling effort is taking place. It is critical that sufficient detail be provided as it happens or shortly thereafter, to allow others not present at the sampling effort, to fully comprehend the procedure and conditions at the time of the sampling effort.

The objective of documenting field activities is to ensure that information regarding the field activities are adequately logged and will be acceptable if it is required as evidence in legal proceedings.

Field team members will keep a field logbook to document all field activities. Field logbooks will provide the means of recording the chronology of data collection activities performed during the investigation. As such, entries will be described in as much detail as possible so that a particular situation could be reconstructed without reliance on memory.

The logbook will be a bound notebook with water-resistant pages. Logbook entries will be dated, legible, and contain accurate and inclusive documentation of the activity. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- The logbook number
- Project name and number
- Site name and location
- Project start date
- End date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, and names of all sampling team members present will be entered. Each page of the logbook will be signed and dated by the person making the entry. All entries will be made in permanent ink, signed, and dated and no erasures or obliterations will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark that is signed and dated by the sampler. The correction shall be written adjacent to the error.

Field activities will be fully documented. Information included in the logbook will include, but may not be limited to the following:

- Chronology of activities, including entry and exit times
- Names of all people involved in sampling activities
- Level of personal protection used
- Any changes made to planned protocol
- Names of visitors to the site during sampling and reason for their visit
- Sample location and identification
- Changes in weather conditions
- Dates (month/day/year) and times (military) of sample collection
- Measurement equipment identification (model/manufacturer) and calibration information
- Sample collection methods and equipment
- Sample depths
- Whether grab or composite sample collected
- How sample composited, if applicable
- Sample description (color, odor, texture, etc.)
- Sample identification code
- Tests or analyses to be performed
- Sample preservation and storage conditions
- Equipment decontamination procedures
- QC sample collection
- Unusual observations
- Record of photographs
- Sketches or diagrams
- Signature of person recording the information

Field logbooks will be reviewed on a daily basis by the Field Team Leader. Logbooks will be supported by standardized forms.

2.2 Analytical Tasks

2.2.1 Analytical SOPs

All method-specific SOPs from the contract laboratory are presented in Attachment 4. A reference table of the SOPs provided follows.

Table 24. CT Laboratory's Standard Operating Procedures (UFP QAPP Worksheet #23)

Category	SOP Title	Date, Revision	SOP #
Anions	Ion Chromatography	10/11/04, Rev 2	CC-IC
Cyanide	Total and Amenable Cyanide	05/04/06, Rev 7	CC-1
Explosives	Semi-Volatile Explosives by Method 8330 with extended analyte list	03/18/08, Rev 1	8330B
Flashpoint	Flashpoint by Pensky-Martens Closed Cup Tester	03/22/02, Rev 1	CC-37
Hexavalent Chromium Liquids	Hexavelent Chromium, Automated Colorimetric	08/21/00, Rev 1	ACOE-CC-34
Hexavalent Chromium Solids	Hexavalent Chromium, Alkaline Digestion Followed by Colorimetric Analysis	05/26/00, Rev 0	ACOE-CC-34B
ICP Metals	Inductively Coupled Plasma Emission – ICP-OES 6000 Series	03/18/08, Rev 0	6105B-6000
	Graphite Furnace Atomic Absorption	03/21/06, Rev 7	6110B
Mercury	Mercury Cold Vapor Atomic Absorption	07/06/07, Rev 8	6120B
Nitrocellulose (Army Method)	Determination of Nitrocellulose in Soil Samples by Basic Hydrolysis and Ion Chromatography of Nitrate and Nitrite	2005, Rev 10	--
PCBs in Soil and Water	Semi-Volatile PCB as Aroclor Analysis	03/14/08, Rev 8	8082
Perchlorate in Water	Determination of Perchlorate in Liquids and Solids Using HPLC/ESI/MS/MS	01/05/09, Rev 1	448
	Method Implementation of EPA Method 6850 Perchlorate by HPLC/ESI/MS/MS	01/17/08	Supplemental document
Pesticides in Soil and Water	Semi-Volatile Organochlorine in Pesticide Analysis	03/03/08, Rev 6	8081
pH of Solids and Wastes	Soil and Waste pH – Electrometric Measurement	02/06/09, Rev 3	CC-24B
Prep for ICP Dissolved Metals	Acid Digestion of Waters for Dissolved or Total Recoverable Metals	07/30/04, Rev 5	6205B
Prep for ICP Metals in Solids	Acid Digestion of Solids and Semi-Solids for Total Metals	10/05/07, Rev 4	6230B
Prep for ICP Total Metals	Total Metals Sample Preparation for FLAA and ICP	10/05/07, Rev 7	6225B
Reactive Cyanide Distillation	Reactive Cyanide Distillation	04/25/06, Rev 0	CC-RCN-Dist
Reactive Cyanide Screen	Reactive Cyanide Pre-Test	04/02/06, Rev 0	CC-RCN-Screen

Table 24. CT Laboratory's Standard Operating Procedures (UFP QAPP Worksheet #23)

Category	SOP Title	Date, Revision	SOP #
Semivolatiles in Soil and Water	Analysis of Semivolatile Organic Compounds by GCMS 8270	07/20/07, Rev 7	8270
	Analysis of PAHs by GC/MS SIM	10/28/08, Rev 2	8270 SIM PAHs
TCLP and SPLP Extractions for Non-Volatiles	TCLP and SPLP Extractions for Non-Volatile Parameters	09/20/04, Rev 4	CL-8B
TCLP and SPLP Extractions for Volatiles	TCLP and SPLP Extractions for Volatile Organic Compounds	08/20/02, Rev 1	CL-8A
Volatiles in Soil and Water	Analysis of Volatile Organic Compounds by GCMS	03/28/08, Rev 9	5280B

2.2.2 Analytical Instrument Calibration Procedure

Analytical instrument calibration procedures are tabulated in the contract laboratory's QA Manual (presented in Attachment 2) and in the respective Analytical SOP (referenced above and presented in Attachment 4).

Calibration procedures for a specific laboratory instrument will consist of initial calibrations, initial calibration verifications, and/or continuing calibration verification. These procedures are as required in the respective analytical methodologies. The initial calibration associated with all analyses must contain a low-level calibration standard which is less than or equal to the reporting limit.

2.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures

Analytical instrument and equipment maintenance, testing, and inspection procedures are tabulated in the contract laboratory's QA Manual (Attachment 2) and in the respective Analytical SOP (referenced above and presented in Attachment 4).

2.2.4 Analytical Supply Inspection and Acceptance Procedures

Materials and consumables will be inspected by the Laboratory Manager against the purchase order specifications to verify their fitness for use. All materials received will be properly labeled and recorded on an inventory log for accuracy. Expiration dates will be assigned to all standards, reagents, and solvents.

Documentation concerning the quality of materials used by the laboratory will be retained at the laboratory. The contract laboratory's QA Manual is presented in Attachment 2, and further describes the analytical supply and acceptance procedures.

2.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures

2.3.1 Sample Collection Documentation

Immediately upon collection, each sample will be labeled with a pre-printed adhesive label, which includes the date and time of collection, sampler's initials, tests to be performed,

preservative (if applicable), and a unique identifier. The following identification scheme will be used:

A. The sample ID number will include the soil, sediment, wastewater, or monitoring well location, along with the sample date and sample interval (if applicable) at which it was collected. The first two letters will represent the sample type:

SS Surface Soil
SD Sediment
SW Surface Water
SB Soil Boring
MW Monitoring Well
WW Waste

The next two numbers represent the site number.

50 SVAD-050

The next two digits represent the location number.

01 i.e. Soil Boring 1

Duplicate and Field QC Blanks will have QC test and flagging codes to identify duplicate environmental and field QC blank samples:

D Field Duplicate
R Equipment Rinsate Blank
T Trip Blank
F Field Blank

The example below represents a duplicate sample from monitoring well MW-1 located on SVAD-050.

Example:
MW-50-01-D

B. The job number will be the number assigned to the particular site.

Example: 3101

C. The analysis required will be indicated for each sample.

Example: SVOC

D. Date taken will be the date the sample was collected, using the format: MM-DD-YY.

Example: 6/22/09

E. Time will be the time the sample was collected, using military time.

Example: 14:30

F. The sampler's name will be printed in the "Sampled By" section.

Example sample labels are presented below:

Job No:	3101-SAD
Client:	ERT
Sample ID:	MW-50-01
Matrix:	Soil
Date Taken:	6/22/09
Time Taken:	14:30
Sampler:	John Doe
Analysis:	SVOC

Job No. _____
Client: _____
Sample Number _____
Date _____ Sample Time _____
Sample Matrix _____
Grab or Composite (explain) _____
Preservatives _____
Analyses _____
Sampler Signature _____

This sample label contains the authoritative information for the sample. Inconsistencies with other documents will be settled in favor of the vial or container label unless otherwise corrected in writing by the field personnel collecting samples or the ERT ITR Officer.

2.3.2 Sampling Handling and Tracking System

Table 25. Sample Handling System (UFP QAPP Worksheet #26)	
Sample collection, packaging, and shipment	
Sample Collection	Field Personnel, ERT
Sample Packaging	Field Personnel, ERT
Coordination of Shipment	Field Personnel, ERT
Type of Shipment	FedEx, Overnight
Sample receipt and analysis	
Sample Receipt	CT Laboratories, Pat Letterer
Sample Custody and Storage	CT Laboratories, Pat Letterer
Sample Preparation	CT Laboratories, Pat Letterer
Sample Analysis	The samples will be analyzed by the individual divisions of the lab depending on each matrix and/or analyte. The Lab Manager (above) will assign tasks to the appropriate personnel at that time.
Sample archiving	
Field Sample Storage	Samples will be stored within the proper storage conditions as soon as possible after sampling. The samples will then be maintained at the proper storage conditions until shipment which will occur as soon as possible. The storage and shipment of samples will be done as quickly as possible and in compliance with appropriate SOPs. The storage and shipping of samples will be done as to allow the laboratories enough time not exceed their holding times. In general, samples will be sent to the laboratories overnight and on a daily basis to minimize field storage time.
Sample Extract/Digestate Storage	Sample extraction/digestion will be performed by CT Laboratories within the holding times of the analytical methods (see relevant preparatory methods in Laboratory SOPs, Attachment 4).
Sample disposal	
Personnel/Organization	CT Laboratories, Pat Letterer
Number of Days from Analysis	Sample disposal will comply with laboratory protocols.

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. ERT's SOP, Packaging and Shipment, was developed to establish packaging and shipping requirements and guidelines for environmental sample shipping. The term "Environmental Sample" refers to any sample that has less than reportable quantities of any hazardous constituents according to Department of Transportation (DOT) 49 CFR - Section 172.

Sample Shipment Materials

- Coolers with return address of ERT office written on inside lid
- Heavy-duty plastic bags
- Plastic zip-top bags, small and large
- Plastic electrical tape
- Fiber tape
- Duct tape
- Vermiculite and/or packing peanuts
- Bubble Wrap (optional)
- Ice
- COC seals
- Completed COC record or CLP custody records if applicable
- Completed Bill of Lading

Sample Shipment Procedures

The following steps must be followed when packing for shipment other than by laboratory provided courier:

1. Select a sturdy cooler in good repair. Secure and tape the drain plug (inside and outside) with fiber or duct tape.
2. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly.
3. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three volatile organic analysis (VOA) vials may be packed in one bag. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.
4. Place two to four inches of packing peanuts or vermiculite into the bag in the cooler and then place the bottles and cans in the bag with sufficient space to allow for the addition of more packing peanuts or vermiculite between the bottles and cans.
5. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place these ice bags on top of, or between, the samples. Fill all remaining space between the bottles or cans with packing peanuts or vermiculite. Securely fasten the top of the large garbage bag with tape (preferably duct tape).

6. Place the completed COC Record or the Contract Laboratory Program (CLP) Traffic Report Form (if applicable) for the laboratory into a plastic zip-top bag, tape the bag to the inner side of the cooler's lid, and then close the cooler.
7. Fiber tape shall be wrapped around each end of the cooler two times, and completed COC seals affixed to the top opposite sides of the cooler half on the fiber tape so that the cooler cannot be opened without breaking the seal. Complete two more wraparounds with fiber tape; place clear tape over custody seals.
8. The shipping containers must be marked "THIS END UP" and arrow labels which indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of the container used to transport environmental samples and shall not be used.
9. Prior to relinquishing the samples to the analytical laboratory, the Site manager will verify that appropriate sample, sample media, sample volumes, sample packaging and a accurately completed COC accompany the samples.

2.3.3 Sample Custody

COC procedures document the handling of each sample from the time it is collected through transportation, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in the field and laboratory. COC procedures will be implemented so that a record of sample collection, transfer of samples between personnel, sample shipping, and receipt by the laboratory is maintained. Records including the cleanliness of sample containers, container shipment from the laboratory to the site, and security of empty containers at the site will be maintained. The COC record serves as a legal record of possession of the sample.

Chain-of-custody (COC) records are initiated by the samplers in the field. The field portion of the custody documentation should include: (1) the project name; (2) signatures of samplers; (3) the sample number, date and time of collection, and whether the sample is grab or composite; (4) signatures of individuals involved in sampling; and (5) if applicable, air bill or other shipping number. An example COC Form is provided in the CT QA Manual (Attachment 2).

On a regular basis (daily or on such a basis that all holding times will be met), samples will be transferred to the custody of the respective laboratories, via third-party commercial carriers or via laboratory courier service.

Sample Custody

Custody is one of several factors that are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files.

A sample or evidence file is considered to be under a person's custody if

- the item is in the actual possession of a person

- the item is in the view of the person after being in actual possession of the person
- the item was in the actual physical possession of the person but is locked up to prevent tampering
- the item is in a designated and identified secure area

Field Custody Procedures

Samples will be collected following the sampling procedures documented in Section 2.1 of this Plan. Documentation of sample collection is described in Section 2.1.2.6 of this Plan. Sample chain-of-custody and packaging procedures are summarized below. These procedures will ensure that the samples will arrive at the laboratory with the chain-of-custody intact.

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or dispatched properly. Field procedures have been designed such that as few people as possible will handle the samples.
- All bottles will be identified by the use of sample labels with sample numbers, sampling locations, date/time of collection, and type of analysis. The sample numbering system is presented in Section 2.3.1 of this Plan.
- Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample label because the pen would not function in wet weather.
- Samples will be accompanied by a properly completed COC form. The sample numbers and locations will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents the transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage location.
- All shipments will be accompanied by the COC record identifying the contents. The original record will accompany the shipment, and copies will be retained by the sampler and placed in the project files.
- Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The custody seals will be attached to the cooler and covered with clear plastic tape after being signed by field personnel. The cooler will be strapped shut with strapping tape in at least two locations. An example COC Seal is presented as Figure 7.

Figure 8. Chain Of Custody Seal

- If the samples are sent by common carrier, the air bill will be used. Air bills will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody forms since the custody forms will be sealed inside the sample cooler and the custody seals will remain intact.
- Samples remain in the custody of the sampler until transfer of custody is completed. This consists of delivery of samples to the laboratory sample custodian, and signature of the laboratory sample custodian on COC document as receiving the samples and signature of sampler as relinquishing samples.

Laboratory Custody Procedures

Samples will be received and logged in by a designated sample custodian or his/her designee. Upon sample receipt, the sample custodian will

- Examine the shipping containers to verify that the custody tape is intact.
- Examine all sample containers for damage.
- Determine if the temperature required for the requested testing program has been maintained during shipment and document the temperature on the COC records.
- Compare samples received against those listed on the COC.
- Verify that sample holding times have not been exceeded.
- Examine all shipping records for accuracy and completeness.
- Determine sample pH (if applicable) and record on COC forms.
- Sign and date the COC immediately (if shipment is accepted) and attach the air bill.
- Note any problems associated with the coolers and/or samples on the cooler receipt form and notify the Laboratory Project Manager, who will be responsible for contacting the ERT Technical Task Manager.
- Attach laboratory sample container labels with unique laboratory identification and test.
- Place the samples in the proper laboratory storage.

Following receipt, samples will be logged in according to the following procedure:

- The samples will be entered into the laboratory tracking system. At a minimum, the following information will be entered: project name or identification, unique sample numbers (both client and internal laboratory), type of sample, required tests, date and time of laboratory receipt of samples, and field ID provided by field personnel.
- The Laboratory Project Manager will be notified of sample arrival.

The completed COC, air bills, and any additional documentation will be placed in the final evidence file.

2.4 Quality Control Samples

2.4.1 Sampling Quality Control Samples

The following presents information on the types and quantities of QA/QC samples to be collected from soil, sediment, groundwater, and surface water. Tables 5 and 11 also present sampling quality control summaries.

- Field Duplicate is a sample collected from the same location as the "parent" sample to assess precision. A field duplicate will be collected one per every 10 samples collected. For aqueous samples, a field duplicate is collected by alternating between the "parent" and field duplicate sample bottles during the filling of the bottles for each analysis. The field duplicate sample will be recorded with the "parent" sample identification code on the well purging and sampling record form.
- Trip Blank is a sample of de-ionized water and Hydrochloric Acid (HCl) prepared by the laboratory and transported with the VOC sample containers. The purpose of the trip blank is to assess whether cross-contamination of VOCs is occurring during transit. The trip blank will be analyzed for VOCs only. The trip blank is kept with the VOC containers from the time they leave the laboratory until they return to the laboratory. Trip blanks must return to the laboratory with the same set of bottles they accompanied to the field. Only one trip blank is needed for each shipment of VOC samples. The trip blank is required to be shipped in the same cooler as the VOC sample containers. It is estimate that there will be one trip blank for every five sample locations.
- Rinsate/Equipment Blanks verify that the equipment used to collect the samples do not cross contaminate other samples, and to verify that equipment decontamination is successful. Rinsate blanks are collected by pouring clean, de-ionized water over the equipment (after decontamination) and collecting it in appropriate sample containers for the same analytes as submitted for laboratory analysis. Rinsate blanks are not collected between each time the equipment is decontaminated, but generally once a day.
- MS/MSD samples will be collected and analyzed to ensure recovery efficiency and to detect potential matrix impediments for the analytical method utilized by the laboratory to obtain analytical results. Matrix Spike samples will be field samples aliquots that are spiked in the laboratory with compounds present on the target analyte(s) list and analyzed under the same conditions as typical field samples. A MSD sample, similar to a duplicate field sample, will be prepared in the same fashion as the Matrix Spike sample and analyzed to ensure reproducibility of results for the particular analytical method.

In addition to the field QC, internal QA checks are performed by the laboratory to verify the quality of the measurements of laboratory investigations and associated tasks.

2.4.2 Analytical Quality Control Samples

The CT Laboratories QAM (Attachment 2) identifies the internal analytical quality control procedures to be utilized. At a minimum, the list below will be included. Also presented in the CT Laboratories QAM are the acceptance criteria for each QC sample type.

- Organics by gas chromatography/mass spectroscopy (GC/MS)
  - Method blanks
  - Initial calibration
  - RLs
  - Continuing calibrations (CCV)
  - MS/MSD
  - LCS
  - IS area count
  - Retention time
  - Instrument performance checks
  - Surrogate recovery
- Organics by GC/high performance liquid chromatography (HPLC)
  - Method blank
  - Initial calibration
  - RLs
  - CCV summary
  - MS/MSD
  - LCS
  - Retention time
  - Performance evaluation mixture
  - Column comparison
  - Surrogate recovery
- Inorganics
  - Method blanks
  - Initial calibration
  - RLs
  - Initial calibration blanks (ICB) and continuing calibration blanks (CCB)
  - CCV
  - MS/MSD
  - Post digestion spike
  - LCS
  - Laboratory duplicates
  - Interference check
  - Serial dilution
  - Retention time

2.5 Data Management Tasks

2.5.1 Project Documentation and Records

To ensure data integrity, defensibility, and retrieval, project data and information will be documented, tracked, and managed, from generation in the field to final use. The primary data management activities include:

- Data transfer from field and laboratory activities to a project filing system
- Data management to ensure that data are stored and output in a manner that continues the chain-of-custody
- Requirements review to ensure that plans for data collection were fulfilled
- Analytical data validation which will report the usability of the data to be used for delineation of vertical and horizontal extent of contamination
- Reporting functions may include outputting data for report tables, statistical analysis, interpretation of data, and electronic transfer.

The following list of documents and records will be generated and kept on file at ERT Headquarters for at least 5 years. Before disposal, records will be offered to USEPA.

Table 26. Project Documents and Records (UFP QAPP Worksheet #29)

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Health and Safety Plan	Field screen records	Case narrative	Data verification checklists	None
Sampling and Analysis Plan	Geophysical investigation records	Definition of laboratory qualifiers	Data validation reports	
Quality Assurance Project Plan	Field sampling audit checklists	Documentation of corrective action results	Data validation checklists	
Field data collection sheets		EDDs		
Field log books		Identification of QC samples		
Chain-of-custody records		Laboratory name		
Airbills		Laboratory sample id numbers		
Communication logs		Quantitation data		
Communication pathways		NELAP accreditation		
Field sampling audit checklists		Reporting forms		
		Sample chronology		
		Tabulated data summary		
		Laboratory data review checklists		

Examples of the field sampling, data verification, and data validation checklists are presented in Attachment 5. The Laboratory's Analytical SOPs, presented in Attachment 4, contains copies of the laboratory's data review checklists.

2.5.2 Data Package Deliverables

Full comprehensive data packages (results and all supporting documentation) will be produced by standard laboratory instrumentation using USEPA-approved methodology. These methods provide low detection limits, defined and potentially rigorous QC, a wide range of calibrated compounds and analytes, matrix recovery and homogeneity information, laboratory process control information, and a fully acceptable level of documentation. Laboratory deliverables will include all applicable QC information and raw data packages to resolve any outstanding QC issues determined during the data validation process. These data can be used for all phases of this project.

A compact disc-read only memory (CD-ROM) containing Adobe Portable Document Format of the complete data validatable reporting package including all raw data shall be submitted by the analytical laboratory.

The approved laboratory will also provide the chemical analysis results as electronic data deliverable (EDDs). For the purpose of having a uniform delivery format for analytical data from laboratories performing environmental analyses, and to ensure that this data can be reviewed in a timely manner prior to use, the USACE (under the FUDS Program) requires implementation of the Staged Electronic Data Deliverable/ Automated Data Review (SEDD/ADR) process.

Data are received from laboratories in the standardized SEDD format based on eXtensible Markup Language (XML). XML is an open, non-proprietary data exchange standard recommended by the World Wide Web Consortium (WC3). Since SEDD is agency and program neutral, the XML data files created by SEDD can easily be shared among various parties. The SEDD XML files are also ideal for long-term data storage since they are non-proprietary.

All analytical services will be provided by CT Laboratories. The contracted data package turnaround time will be 30 days.

2.5.3 Data Reporting Formats

Measurements and sample collection information will be transcribed directly into easily identifiable field logbook or onto standardized forms. If errors are made, results will be legibly crossed out, initialed and dated by the person recording the data, and corrected in a space adjacent to the original (erroneous) entry. Daily reviews of the field records by the Field Team Leader will ensure that:

- Logbooks and standardized forms have been filled out completely and that the information recorded accurately reflects the activities that were performed.
- Records are legible and in accordance with good record keeping procedures, i.e., entries are signed and dated, data are not obliterated, changes are initialed, dated, and explained.
- Sample collection, handling, preservation, and storage procedures were conducted in accordance with the protocols described in the Plan, and that any deviations were documented and approved by the appropriate personnel.

A more concise description of ERT's documentation procedures is presented in *Use of Field Logbooks* (Attachment 3).

The laboratory will report data in a format that will allow raw data validation to take place; this report will be equivalent to Level IV with Contract Laboratory Program (CLP)-like data package deliverables. This means that the sample results should be able to be re-created from the data presented in the package.

The raw data required will include laboratory instrument printouts of chromatograms and mass spectra, calibration records, sample preparation records, spiking information, and dilution records. In instance when manual integration is necessary, chromatograms before and after each manual integration and a reason for manual integration will be provided in the data packages. In addition, the laboratory will present reports that will contain enough information for flagging the

data according to data qualifier flags, including date prepared, date analyzed, sample results, dilution factors, spike levels and percent recoveries, calibration summaries, blank results, and laboratory duplicate results. All surrogate recoveries should be reported including recoveries for QC samples and field samples.

The deliverables package will include, at a minimum, a cover page (with laboratory name and address, ERT project name, and ERT project number), table of contents referencing individual sections in the data package, original chain-of custody (COC) forms, shipping documents, sample cooler receipt form (including cooler temperature), cross-reference table, analytical results, a copy of all corrective action reports, organics, inorganic and general chemistry data packages, internal laboratory QC/QA data with their respective acceptance criteria, and a case narrative documenting the resolution of all corrective actions and noncompliance events. All ERT samples will be clearly referenced to the associated QC samples.

The analytical results for each sample will contain the following information at a minimum:

- Project name and project number
- Field sample identification number as written on the chain-of-custody
- Laboratory name
- Laboratory sample identification number
- Date sample collected
- Date sample received
- Date sample extracted or prepped
- Date sample analyzed
- Analysis time when analysis holding time is less than 48 hours
- Preparation and analysis batch numbers
- Method numbers for all preparations, cleanup procedures and analyses
- Analyte
- Reporting limit adjusted for sample-specific factors (e.g., aliquot size, dilution or concentration factor, moisture content)
- Analytical result (solid matrices must be reported on a dry weight basis)
- Concentration units
- Dilution factor
- Matrix
- Percent moisture or percent solids
- Sample aliquot analyzed
- Final extract volume.

The internal laboratory QA/QC information shall be provided. These data shall include the quantitation limits (QLs) for the project-specific analytes and correlate the method QC data with the corresponding environmental samples on a per-batch basis. The method QC data includes all

spike recoveries, all measures of precision and accuracy, including relative percent differences (RPDs), and all control limits for precision and accuracy. This will include laboratory performance information such as results for method blanks, initial and continuing calibration information, recoveries for Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) samples, RPD for LCS/LCSD pairs, recoveries of QC sample surrogates; and matrix-specific information such as sample duplicate RPDs, Matrix Spike (MS) and Matrix Spike Duplicate (MSD) recoveries, MS/MSD RPDs, serial dilution percent differences, interference check sample results, and field sample surrogate recoveries. Any deviations from the control limits (Attachment 2) shall be noted.

All relevant raw data and documentation, including (but not limited to) logbooks, data sheets, electronic files, final reports, etc., will be maintained by the laboratory for at least 5 years. ERT will be notified in writing 30 days before disposal of any relevant laboratory records.

2.5.4 Data Handling and Managment

All analytical data will be compiled by the Laboratory Information Management System (LIMS) and sent by the Laboratory Manager to the ERT Project Manager by email and standard mail (CD and hard copy data) within 30 days after sample submittal. All relevant raw analytical data and documentation, including (but not limited to) logbooks, data sheets, electronic files, final reports, etc., will be maintained by the laboratory for at least 5 years. ERT will be notified in writing 30 days before disposal of any relevant laboratory records.

Upon receipt of analytical data packages, the ERT Project Chemist will review the deliverables for completeness and produce a data verification checklist (example available upon request). Once data have been verified, they will be uploaded to the EDS ftp site for validation. All analytical data will then be validated bu EDS. and a data validation report will be produced within 30 days after receipt. ERT will be notified of completion of the validation report, and reports will become retrievable from the EDS ftp site.

All records received by ERT will be kept on file at ERT Headquarters for at least 5 years.

2.5.5 Data Tracking and Control

Hard copies and digital information produced by this investigation will be kept and organized by the ERT Project Manager at ERT's Annapolis Junction office for at least five years. All records will be offered to Mr. Thomas Barounis of USEPA Region 5 prior to disposal.

3.0 ASSESSMENT/OVERSIGHT

3.1 Assessments and Response Actions

3.1.1 Planned Assessments

The entire sampling program will be under the direction of ERT Internal Technical Review (ITR). The emphasis in this program is on preventing problems by identifying potential errors, discrepancies, and gaps in the data-collection-laboratory-analysis-interpretation process. Any problems identified will be promptly resolved. Likewise, follow-up corrective action is always an option in the event that preventative corrective actions are not totally effective.

The acceptance limits for the sampling and analyses to be conducted in this program will be those stated in the method or defined by other means in the QAPP. Corrective actions are likely to be immediate in nature and most often will be implemented by the contracted laboratory analyst or the ERT Project Manager. The corrective action will usually involve recalculation, reanalysis, or resampling. Planned project assessments are listed in Table 27.

3.1.2 Assessments and Findings and Corrective Action Responses

Deficiency tracking and corrective actions will be taken whenever there is a deficiency in characteristic(s), documentation, or procedures that result in the quality of an item being deemed unacceptable or indeterminate with respect to specified criteria. Nonconformance examples include test failure, physical defects, data losses, deviations from prescribed project plans, and deviations from industry-accepted processes and procedures. If nonconformance is related to a hazardous condition or potential safety concerns, corrective action will immediately be taken and documentation will be completed as an immediate follow-up. Prompt corrective action for safety issues will never be impeded by the need to process a deficiency report.

Corrective action will be initiated when potential or existing conditions are identified that may adversely impact data quality or quantity. It is the responsibility of the individual who first recognizes an out-of-control event to initiate corrective action and to document the action. Notification of the nonconformance will be directed to the individual's supervisor. It is the supervisor's responsibility to coordinate corrective action with the Project Manager and to follow up to ensure re-establishment of control. Events that require corrective action include:

- Violation of established field or sample handling procedures
- Violation of established analytical controls
- Violation of established field or sample collection procedures
- Violation of established sample shipping or receiving procedures
- Results of performance, system, or project QA audits
- Data reporting errors.

Table 27. Planned Project Assessments (UFP QAPP Worksheet #31)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Title of Person Responsible for Assessment	Title of Person Responsible for Responding to Assessment Findings	Title of Person Responsible for Implementing Corrective Action (CA)	Title of Person Responsible for Monitoring CA Effectiveness
Field Sampling TSA	Once per Media	Internal	ERT	Project QA/QC Manager	Field Manager	Project Manager	Project Manager
Laboratory TSA	Per Laboratory QA Manual	External	CT Laboratories	Laboratory QA Manager	Project Chemist	Project Manager	Project Manager
Data Review TSA	Once per Data Package	External	EDS	Senior Chemist	Project Chemist	Project Manager	Project Manager
Mangagement Systems Review	Quarterly Review	Internal	ERT	Project QA/QC Manager	Project Manager	Project Manager	Project Manager

TSA Technical Systems Audit

When a deficiency is detected, a deficiency report will be generated, describing and documenting the occurrence and the corrective action taken or suggested to be taken. The appropriate criteria will be cited and sufficient supporting data will be provided to allow a resolution to the deficiency. The report will be generated by the Site Manager, and will be provided to the Project Manager for review and approval of the proposed corrective action. The report will describe the cause for the deficiency, corrective action taken, measures taken to prevent a recurrence, and the date actions are to be completed. The PM will review and sign the deficiency report.

The deficiency will be reported to the Contracting Officer's Representative (COR) within 48 hours. Correction of the deficiency will be verified and the deficiency report indicating closeout will be signed by the Project Manager. The deficiency report will not be closed out until all corrective and preventive measures have been completed.

The Project Manager will maintain a deficiency log of all open deficiencies, date of issue, responsible organization or individual, date of anticipated corrective action, and closed-out deficiency reports. The log will provide the deficiency number, a brief description of the action, and the date closed. Records to be retained in the project file will contain pertinent information necessary to document resolution of the deficiency including scope and significance of the problem.
Corrective action may take several forms, but the following steps are almost always included:

- Check the calculations
- Check the instrument for proper setup
- Re-analyze the control item
- Stop work (if necessary).

The corrective action may be immediate or long term. A corrective action requiring immediate response may be recalibration, recalculation, reanalysis, or repeating sample collection. Long-term corrective action may be identified through, but not limited to, performance evaluation samples, standards, and control charts. Corrective actions will be documented on a Field Change Report and maintained in project files.

The Field Change Report includes a description of the corrective action planned and has space for follow-up. The Project Manager will verify that initial action has been taken and appears to be effective and, at an appropriate later date, will check to see if the problem has been resolved. The Project Manager receives a copy of all Field Change Reports and enters them into the Corrective Action Log. This permanent record will aid the Project Manager in following up and will assist in resolving QA problems. An example Field Change Request form is included in the SAP.

Corrective action in the field may be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the Plan), or when sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. The field team may identify the need for corrective action. The Site Manager will approve the corrective action and notify the ERT Project Manager. The ERT Project Manager will approve

the corrective measure. The Site Manager will ensure that the corrective measure is implemented by the field team.

Corrective actions will be implemented and documented in the field record book. Documentation will include:

- A description of the circumstances that initiated the corrective action
- The action taken in response
- The final resolution
- Any necessary approvals

No staff member will initiate corrective action without prior communication of findings through the proper channels.

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, omissions or discrepancies with chain-of-custody documentation, low/high pH readings, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with laboratory analysts and Laboratory Section Leaders, it may be necessary for the Laboratory QA Manager to approve the implementation of corrective action. The laboratory SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain QC criteria are not met, loss of sample through breakage or spillage, etc.

The analyst may identify the need for corrective action. The Laboratory Section Leader, in consultation with the staff, will approve the required corrective action to be implemented by the laboratory staff. The Laboratory QA Manager will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, the ERT ITR will be notified. The ERT ITR will notify the ERT Project Manager, who in turn will contact all levels of project management for concurrence with the proposed corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action files, and the narrative data report sent from the laboratory to the ERT Project Manager. If the corrective action does not rectify the situation, the laboratory will contact the ERT Project Manager, who will determine the action to be taken and inform the appropriate personnel. If potential problems are not solved as an immediate corrective action, the contractor will apply formalized long-term corrective action, if necessary.

3.2 QA Management Reports

Periodic QA management reports will be producted to ensure that all parties are updated on project status and results of all QA assessments. Efficient communication of project status and problems in this manner will allow project managers to implement timely and effective corrective actions so data generated can meet PQOs. Table 28 summarizes the QA Management Reports planned for this RI/FS.

Table 28. QA Management Reports (UFP QAPP Worksheet #33)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Progress Report	Monthly	15th of each month	ERT Project Manager	USACE-Louisville
Eng Form 93-1	Monthly	15th of each month	ERT Project Manager	USACE- Louisville
Field Reports	Daily, Weekly	End of each day during field activities, weekly summary prepared by the following month	ERT Site and Project Manager	USACE- Louisville
Corrective Action Reports	As needed	Within 48 hours of discovery.	ERT Site and Project Manager, ERT ITR	USACE- Louisville
Data Validation Report	Per sampling event	30 days after receipt of laboratory data	EDS	ERT
Data Validation Report Summary	Per sampling event	15 days after receipt of laboratory data validation report	ERT Project Chemist and Project Manager	USACE- Louisville
Data Usability Report	Per sampling event	30 days after receipt of laboratory data validation report	ERT Project Chemist and Project Manager	USACE- Louisville
Other Types of Reports are presented throughout the QAPP				

3.3 Final Project Report

The final project report will include the following information in the QA/QC section or as attachments, as deemed appropriate:

- Summary of project QA/QC programs and trainings conducted during the project
- Conformance of project activities to QAPP requirements and procedures
- Status of project and schedule delays
- Deviations from the approved QAPP and approved amendments to the QAPP
- Results and trends of PT samples performed by all laboratories (per analytical group, matrix, and concentration level)
- Description and findings of TSAs and other assessments
- Results of data review activities in terms of amount of usable data generated
- Required corrective actions and effectiveness of corrective action implementation
- Data usability assessments in terms of precision, accuracy, representativeness, completeness,
- comparability, and sensitivity (refer to Section 5.2)
- Limitations on the use of measurement data generated

In addition, the final report will include the following information:

- Narrative and timeline of project activities
- Summary of PQO development
- Reconciliation of project data with PQOs
- Summary of major problems encountered and their resolution
- Data summary, including tables, charts, and graphs with appropriate sample identification or station location numbers, concentration units, percent solids (if applicable), and data quality flags
- Conclusions and recommendations

4.0 DATA REVIEW

4.1 Overview

Review of field and analytical data will undergo a sequential three-step process to ensure that PQOs are met, including:

1. Verification
2. Validation
3. Usability Assessment

4.2 Data Review Steps

4.2.1 Step I: Verification

Sampling and analysis data will be reviewed for completeness to see if data required for the project are available, and a data verification checklist (verification report) will be completed. Table 29 summarizes the data verification process.

Table 29. Verification (Step I) Process (UFP QAPP Worksheet #34)

Verification Input	Description	Internal/ External	Title of Responsible Individual(s)
Field Data Measurements	Daily field calibration logs and measurement results from the portable multi-meters will be reviewed daily internally for completeness. Any corrective actions will be addressed with the field samples prior to further measurements.	Internal	Field Manger Field Sampling Team
Field Notes	Field notes will be reviewed daily for completeness, accuracy, and comparability between sample locations and field samplers. Any required corrective actions will be addressed with the field samplers prior to further site work. The Field Manager will summarize the results of field notes in a brief daily field summary statement and transmit this brief summary to the ERT Project Manager.	Internal	Field Manager Field Sampling Team
COCs and Shipping Forms	COCs and shipping documentation will be reviewed upon their completion and verified against the labeled and packed samples and coolers that they represent. Upon verification of completeness and accuracy, the reviewer will sign the documents and copies will be retained in the site file.	Internal	Field Manager Field Sampling Team
Sample Receipt	Samples will be reviewed upon receipt by the contract laboratory. COCs will be verified against the labeled samples that they represent. Upon verification of completeness and accuracy, the reviewer will prepare a sample receipt report.	External	Laboratory Technician
Laboratory Data Packages	All laboratory data packages will be verified for internally by the laboratory performing the analyses. Data packages will be reviewed for completeness prior to submittal to ERT. All received data packages will be verified internally according to the data verification procedures.	External Internal	Laboratory Manger ERT Project Chemist
Validation Reports	All validation reports will be reviewed for completeness upon submittal to ERT.	Internal	ERT Project Chemist
Field Audit Reports	These report indicate that the samples were collected, field measurements performed, and field equipment calibrated according to the QAPP.	Internal	ERT Project Chemist

To assist with the data verification process, a review of the project planning documents, sampling documents, and relevant external reports will also be conducted. Once all inputs are deemed present, the analytical data package, it will be forwarded to EDS for data validation, using either ERT's File Transfer Protocol (ftp) web site or put on a CD-ROM and mailed.

4.2.2 Step II: Validation

4.2.2.1 Step IIa Validation Activities

Sampling and analysis data will be assessed for compliance with method, procedure, and contract requirements in the Step IIa Validation process.

4.2.2.2 Step IIb Validation Activities

Analytical data will be assessed for performance of the analytical process by comparing results with the MPC.

Analytical data will be validated by EDS according to *CLP National Functional Guidelines for Organic Data Review* (EPA, 1999) and the *CLP National Functional Guidelines for Inorganic Data Review* (EPA, 2002). Following data validation, reports that include qualified data will be prepared by EDS and submitted to ERT. Table 30 summarizes the validation process, and Table 31 provides a validation summary.

Table 30. Validation (Steps IIa and IIb) Process (UFP QAPP Worksheet #35)

Step IIa/IIb	Validation Input	Description	Organization(s) and Title(s) Primarily Responsible for Validation
IIa	EDDs	Ensures that all required information on sampling and analysis is provided.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	Analytes	Ensures that required list of analytes were reported as specified in QAPP.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	COCs	Examination of traceability of data from time of sample collection until reporting of data. COC examined against contract, method, and/or procedural requirements.	CT Laboratories, Laboratory Manager ERT, Project Chemist
IIa	Holding time and sample preservation	Ensures that samples were analyzed within required holding times and preserved specific to analysis requirements and were within required temperature range.	Analytical laboratory for most data, EDS will perform validation on subcontracted laboratory data
IIa	Sample Handling	Ensures that required sampling handling, receipt, and storage procedures were followed, and that any deviations were documented.	CT Laboratories, Laboratory Manager ERT, Project Chemist
IIa	Sampling Methods and Procedures	Establishes that required sampling methods were used and that any deviations were noted. Ensures that the sampling procedures and field measurements met performance criteria and that any deviations were documented.	ERT, Field Manager ERT, Project Chemist
IIa	Analytical Methods and Procedures	Establishes that required analytical methods were used and that any deviations were noted. Ensures that QC samples met performance criteria and that any deviations were documented,	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	Manual Integrations	Determines that peak(s) are correctly integrated and concentrations reported are correct by calculation.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	Post Digestion Spikes	Determines if matrix effects may be a factor in the results. The spike addition shall produce a method-specified minimum concentration above the method reporting limit. For inorganic compounds, a post-digestion spike is a spike added to the sample after preliminary preparation, usually just before analysis. The post-digestion spike technique is normally used when an MS fails. Therefore, the sample spiked would be that sample on which an MS was originally performed. The post-digestion spike provides the analyst with information regarding matrix-related interferences on the analytical system that may still be present in the sample following digestion. This technique is typically used for ICP spectrometry analysis but is appropriate to other techniques as well.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist

Table 30. Validation (Steps IIa and IIb) Process (UFP QAPP Worksheet #35)

Step IIa/IIb	Validation Input	Description	Organization(s) and Title(s) Primarily Responsible for Validation
IIa/IIb	Data Qualifiers	Determines that laboratory data qualifiers were defined and applied as specified in methods, procedures, or contracts. Determines that any deviations are justified.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	Proficiency Testing (PT)	Confirms acceptance of PT sample results against performance requirements as specified in methods, procedures, or contracts.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa	Recalculations	Checks 10% of all laboratory data by recalculating.	EDS, Senior Chemist
IIa	Standards	Determines that standards are traceable and meet contract, method, or procedural requirements.	CT Laboratories, Laboratory Manager
IIa	Audits	Review field and laboratory audit reports and accreditation and certification records for the laboratory's performance on specific methods.	ERT, Project Manager ERT, Project Chemist
IIb	Analytical Deviations	Determines the impacts of any deviations from analytical methods.	CT Laboratories, Laboratory Manager EDS, Senior Chemist
IIb	Confirmatory Analyses	Evaluates agreement of laboratory results.	CT Laboratories, Laboratory Manager EDS, Senior Chemist
IIb	QA/QC	Evaluates accuracy and precision of results.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIb	Project Quantiation Limits	Determines if quantitation limits (QL) were achieved as outlined in QAPP, and if the laboratory successfully analyzed a standard at the QL.	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIb	Sampling Plan and Procedures	Determines if the Sampling Plan was executed as specified.	ERT, Project Manager ERT, Project Chemist
IIa/IIb	Validation Report	Summarizes deviations from methods, procedures, or contracts. Summarizes outcome of comparison of data to MPC. Report includes qualified data and explanation of all qualified data.	EDS, Senior Chemist

Table 31. Validation (Steps IIa and IIb) Summary (UFP QAPP Worksheet #36)

Step IIa/IIb	Matrices	Analytical Group	Concentration Level	Validation Criteria	Data Validator(s)
IIa /IIb	Soil/Sediment	TCL VOCs	Low Level	USEPA SW 846 Method 8260B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	TCL SVOCs	Low Level	USEPA SW 846 Method 8270C QAPP Attachment 1 and Table 15 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	Pesticides	Low Level	USEPA SW 846 Method 8081A QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	PCBs	Low Level	USEPA SW 846 Method 8082 QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	TAL Metals	Low Level	USEPA SW 846 Method 6010B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	Cyanide	Low Level	USEPA SW 846 Method 9012A QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	Explosives	Low Level	USEPA SW 846 Method 8330B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Soil/Sediment	Nitrocellulose	Low Level	USEPA SW 846 Method 9056M QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	TCL VOCs	Low Level	USEPA SW 846 Method 8260B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	TCL SVOCs	Low Level	USEPA SW 846 Method 8270C QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	Pesticides	Low Level	USEPA SW 846 Method 8081A QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist

Table 31. Validation (Steps IIa and IIb) Summary (UFP QAPP Worksheet #36)

Step IIa/IIb	Matrices	Analytical Group	Concentration Level	Validation Criteria	Data Validator(s)
IIa /IIb	Groundwater/ Surface Water	PCBs	Low Level	USEPA SW 846 Method 8082 QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	TAL Metals	Low Level	USEPA SW 846 Method 6010B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	Cyanide	Low Level	USEPA SW 846 Method 9012A QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	Explosives	Low Level	USEPA SW 846 Method 8330B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	Nitrocellulose	Low Level	USEPA SW 846 Method 9056M QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater/ Surface Water	Anions	Low Level	USEPA SW 846 Method 353.2/ SM4500-NO_2B QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist
IIa /IIb	Groundwater	Perchlorate	Low Level	USEPA SW 846 Method 6050 QAPP Attachment 1 and Table 13 QAPP Attachment 2	CT Laboratories, Laboratory Manager ERT, Project Chemist EDS, Senior Chemist

4.2.3 Step III: Usability Assessment

The usability assessment will consider whether data meet project quality objectives as they relate to the decision to be made, and evaluate whether data are suitable for making that decision. The usability assessment is a data review and will be performed by the project team, and only on data of known and document quality (i.e verified and validated data).

4.2.3.1 Data Limitations and Actions from Usability Assessment

The following data quality indicators (precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity) are important components of validation and usability assessment. A description of how they should be incorporated into the final report is found under each parameter heading.

Precision

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms. QC measures for precision include field duplicates, laboratory duplicates, matrix spike duplicates, analytical replicates, and surrogates.

In order to meet the needs of the data users, project data must meet the measurement performance criteria for precision specified in the PQO and MPC section of the QAPP (section 1.5). Precision may be the result of one or more of the following: field instrument variation, analytical measurement variation, poor sampling technique, sample transport problems, or spatial variation (heterogeneous sample matrices). To identify the cause of imprecision, the field sampling design rationale and sampling techniques will be evaluated by the reviewer, and both field and analytical duplicate/replicate sample results will be reviewed.

If poor precision is indicated in both the field and analytical duplicates/replicates, then the laboratory may be the source of error. If poor precision is limited to the field duplicate/replicate results, then the sampling technique, field instrument variation, sample transport, and/or spatial variability may be the source of error.

If data validation reports indicate that analytical imprecision exists for a particular data set or sample delivery group (SDG), then the impact of that usability must be discussed in the usability report.

Accuracy/Bias

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias), that are due to sampling and analytical operations. Examples of QC measures for accuracy included in this project are proficiency testing (PT) samples, matrix spikes, laboratory control samples (LCSs), and equipment blanks. In order to meet the needs of the data users, project data must meet the measurement performance criteria for accuracy/bias specified in the PQO and MPC section of the QAPP (section 1.5).

Representativeness

Representativeness is the measure of the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition. In order to meet the needs of the data users, project data must meet the measurement performance criteria for sample representativeness specified in the PQO and MPC section of the QAPP (section 1.5).

The QAPP discusses how the QA/QC activities (review of sampling design and SOPs, field sampling TSAs, and analysis audits, etc.) and QC sample data will be reviewed to assess sample representativeness. If field duplicate precision checks indicate potential spatial variability, additional scoping meetings and subsequent resampling may be needed in order to collect data that are more representative of a nonhomogeneous site.

Comparability

Comparability is the degree to which different methods, data sets, and decisions agree or can be represented as similar. Comparability describes the confidence (expressed qualitatively or quantitatively) that two data sets can contribute to a common analysis and interpolation. In order to meet the needs of the data users, project data must meet the measurement performance criteria for comparability specified in the PQO and MPC section of the QAPP (section 1.5).

The QAPP includes methods and formulas for assessing data comparability for each matrix, analytical group, and concentration level.

Sensitivity and Quantitation Limits

Sensitivity is the capability of a test method or instrument to discriminate between measurement responses representing different levels (i.e. concentrations) of a variable of interest. Examples of QC measures for determining sensitivity include laboratory fortified blanks, a method detection limit study, and calibration standards at the quantitation limit (QL). In order to meet the needs of the data users, project data must meet the measurement performance criteria for sensitivity and project QLs specified in the PQO and MPC section of the QAPP (section 1.5).

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct, normal circumstances. In order to meet the needs of the data users, project data must meet the measurement performance criteria for data completeness specified in the PQO and MPC section of the QAPP.

4.2.3.2 Activities

Activities

The entire project team will reconvene to perform the usability assessment to ensure that the PQOs are understood and the full scope is considered. The items listed in Table 33 are examples of specific items that will be considered during the data usability assessment for the SVAD-050 RI/FS.

Table 32. Considerations for Usability Assessment (UFP QAPP Worksheet #37)

Consideration	Usability Assessment
Background	Determine if background levels have been adequately established (if appropriate).
Chain-of-Custody	Establishes that any problems with documentation or custody procedures do not prevent the data from being used for the intended purpose.
Comparability	Ensures that results from different data collection activities achieve an acceptable level of agreement.
Completeness	Evaluates the impact of missing information. Ensures that enough information was obtained for the data to be usable (completeness as defined in PQOs documented in the QAPP).
Critical Samples	Establishes that critical samples and critical target analytes/COPCs, as defined in the QAPP, were collected and analyzed. Determine if the results meet criteria specified in the QAPP.
Damaged Samples	Determines whether the data from damaged samples are usable. If the data cannot be used, determines whether resampling is necessary.
Data Deliverables and QAPP	Ensures that all necessary information was provided, including but not limited to validation results. Determines the impact of deviations on the usability of data.
Data Restrictions	Describes the exact process for handling data that do not meet PQOs (i.e., when measurement performance criteria are not met).
Holding Times	Determines the acceptability of data where holding times were exceeded.
Matrix	Evaluates matrix effects (interference or bias).
Meteorological Data and Site Conditions	Evaluates the possible effects of meteorological (e.g., wind, rain, temperature, flood) and site conditions on sample results. A review of field reports to identify whether any unusual conditions were present and how the sampling plan was executed.
PT Sample Results	Determines the implications of any unacceptable analytes (as identified by the PT sample results) on the usability of the analytical results. Describes any limitations on the data.

Table 32. Considerations for Usability Assessment (UFP QAPP Worksheet #37)

Consideration	Usability Assessment
QC Samples	Evaluates the implications of unacceptable QC sample results on the data usability for the associated samples. For example, will consider the effects of observed blank contamination.
Sampling Locations	Determines if alterations to sample locations continue to satisfy the project objectives.
SOPs and Methods	Evaluates the impact of deviations from SOPs and specified methods on data
Usability Decision	Determines if the data can be used to make a specific decision considering the implications of all deviations and corrective actions.
Usability Report	Discusses and compare overall precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity for each matrix, analytical group, and concentration level. Describes limitations on the use of project data if criteria for data quality indicators are not met.

5.0 REFERENCES

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ATTACHMENT 1
Reference Limits and Evaluation Table

REFERENCE LIMITS AND EVALUTATIONS TABLE
SOLIDS
SOIL & SEDIMENT

VOLATILE ORGANIC COMPOUNDS BY GC/MS (LOW LEVEL)
SW 846 METHOD 8260B
Preparatory SW 846 Method 5035

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**TACO**	**Units**
					Residential Soil		
1,1,1-Trichloroethane	71-55-6	5	0.5	1.7	9,000,000	2,000	µg/kg
1,1,2,2-Tetrachloroethane	79-34-5	5	1.1	3.5	590	NS	µg/kg
1,1,2-Trichloroethane	79-00-5	5	0.6	1.9	1,100	20	µg/kg
1,1-Dichloroethane	75-34-3	5	0.3	1.0	3,400	23,000	µg/kg
1,1-Dichloroethene	75-35-4	5	0.5	1.5	250,000	60	µg/kg
1,2,4-Trichlorobenzene	120-82-1	5	1.6	5.2	180,000	5,000	µg/kg
1,2-Dibromo-3-chloropropane	96-12-8	5	1.1	3.8	6	**2**	µg/kg
1,2-Dichlorobenzene	95-50-1	5	0.9	3.2	2,000,000	17,000	µg/kg
1,2-Dichloropropane	78-87-5	5	0.6	2.1	930	30	µg/kg
1,3-Dichlorobenzene	541-73-1	5	1.2	3.9	NS	NS	µg/kg
1,3-Dichloropropane	142-28-9	5	0.4	1.4	1,600,000	NS	µg/kg
1,4-Dichlorobenzene	106-46-7	5	1.4	4.5	2,600	2,000	µg/kg
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	8.7	2.6	8.7	43,000,000	NS	µg/kg
2-Butanone	78-93-3	21	6	21	28,000,000	NS	µg/kg
2-Hexanone	591-78-6	20	5	15	NS	NS	µg/kg
4-Methyl-2-pentanone	108-10-1	20	2.9	10	5,300,000	NS	µg/kg
Acetone	67-64-1	20	6	19	1,443,616	25,000	µg/kg
Benzene	71-43-2	5	0.3	1.2	61,000,000	30	µg/kg
Bromobenzene	108-86-1	5	0.9	3.0	1,100	NS	µg/kg
Bromochloromethane	74-97-5	5	0.6	2.0	NS	NS	µg/kg
Bromodichloromethane	75-27-4	5	0.4	1.4	10,000	600	µg/kg
Bromoform	75-25-2	5	0.7	2.2	61,000	800	µg/kg
Bromomethane	74-83-9	5	1.2	4.1	7,900	200	µg/kg
Carbon disulfide	75-15-0	5	1.0	3.2	670,000	32,000	µg/kg
Carbon tetrachloride	56-23-5	5	0.7	2.2	250	70	µg/kg
Chlorobenzene	108-90-7	5	0.8	2.7	310,000	1,000	µg/kg
Chloroethane	75-00-3	10	0.4	1.3	15,000,000	NS	µg/kg
Chloroform	67-66-3	5	0.4	1.4	300	300	µg/kg
Chloromethane	74-87-3	5	0.5	1.5	1,700	NS	µg/kg
cis-1,2-Dichloroethene	156-59-2	5	0.5	1.5	780,000	400	µg/kg
cis-1,3-Dichloropropene	10061-01-5	5	0.6	1.9	1,700	5	µg/kg
Dibromochloromethane	124-48-1	10	0.7	2.2	5,800	400	µg/kg
Ethylbenzene	100-41-4	5	0.7	2.3	5,700	13,000	µg/kg
m & p-Xylene	1330-20-7	10	1.7	5.7	9,200,000	150,000	µg/kg
Methyl tert-butyl ether	1634-04-4	5	0.5	1.8	39,000	320	µg/kg
Methylene chloride	75-09-2	10	1.9	6.3	11,000	20	µg/kg
o-Xylene	95-47-6	5	1.0	3.3	5,300,000	190,000	µg/kg
Styrene	100-42-5	5	0.5	1.8	1,700,000	4,000	µg/kg
Tetrachloroethene	127-18-4	5	0.9	3.0	570	60	µg/kg
Toluene	108-88-3	5	0.6	2.1	5,000,000	12,000	µg/kg
trans-1,2-Dichloroethene	156-60-5	5	0.6	2.1	110,000	700	µg/kg
trans-1,3-Dichloropropene	10061-02-6	5	0.8	2.6	1,700	5	µg/kg
Trichloroethene	79-01-6	5	0.5	1.8	2,800	60	µg/kg
Trichlorofluoromethane	75-69-4	5	0.9	3.0	8,000,000	NS	µg/kg
Vinyl chloride	75-01-4	5	0.7	2.4	60	10	µg/kg
Surrogates			**Lower**	**Upper**			**Units**
1,2 Dichloroethane-d4	17060-07-0		70	130			% Recovery
Bromofluorobenzene	460-00-4		85	120			% Recovery
d8-Toluene	2037-26-5		85	115			% Recovery
Dibromofluoromethane	1868-53-7		70	130			% Recovery

REFERENCE LIMITS AND EVALUTATIONS TABLE
SOLIDS
SOIL & SEDIMENT

SEMIVOLATILE ORGANIC COMPOUNDS (LOW LEVEL) + SIM QSM
SW 846 METHOD 8270C
Preparatory SW 846 Method 3540

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**TACO**	**Units**
					Residential Soil		
2,4,5-Trichlorophenol	95-95-4	350	105	350	61,000,000	270,000	µg/kg
2,4,6-Trichlorophenol	88-06-2	260	76	260	44,000	660	µg/kg
2,4-Dichlorophenol	120-83-2	320	93	320	180,000	1,000	µg/kg
2,4-Dimethylphenol	105-67-9	260	77	260	1,200,000	9,000	µg/kg
2,4-Dinitrophenol	51-28-5	710	220	710	120,000	3,300	µg/kg
2,4-Dinitrotoluene	121-14-2	100	23	76	120,000	250	µg/kg
2,6-Dinitrotoluene	606-20-2	100	20	67	61,000	260	µg/kg
2-Chloronaphthalene	91-58-7	100	13	45	6,300,000	NS	µg/kg
2-Chlorophenol	95-57-8	350	110	350	390,000	4,000	µg/kg
2-Methylnaphthalene	91-57-6	4.6	1.38	4.6	310,000	NS	µg/kg
2-Methylphenol	95-48-7	340	110	340	NS	15,000	µg/kg
2-Nitroaniline	88-74-4	100	26	87	NS	NS	µg/kg
2-Nitrophenol	88-75-5	400	120	400	NS	NS	µg/kg
3 & 4-Methylphenol	1319-77-3	350	110	350	NS	NS	µg/kg
3,3'-Dichlorobenzidine	91-94-1	930	280	930	1,100	1,300	µg/kg
3-Nitroaniline	99-09-2	110	32	110	NS	NS	µg/kg
4,6-Dinitro-2-methylphenol	534-52-1	500	71	240	NS	NS	µg/kg
4-Bromophenyl-phenyl ether	101-55-3	100	10	34	NS	NS	µg/kg
4-Chloro-3-methylphenol	59-50-7	310	91	310	240,000	NS	µg/kg
4-Chloroaniline	106-47-8	220	65	220	NS	700	µg/kg
4-Chlorophenyl-phenyl ether	7005-72-3	110	31	110	NS	NS	µg/kg
4-Nitroaniline	100-01-6	110	31	110	NS	NS	µg/kg
4-Nitrophenol	100-02-7	1100	330	1100	NS	NS	µg/kg
Acenaphthene	83-32-9	4.8	1.42	4.8	3,400,000	570,000	µg/kg
Acenaphthylene	208-96-8	4.64	1.39	4.64	NS	NS	µg/kg
Acetophenone	98-86-2	110	32	110	7,800,000	NS	µg/kg
Anthracene	120-12-7	4.98	1.50	4.98	17,000,000	12,000,000	µg/kg
Benzo(a)anthracene	56-55-3	4.9	1.44	4.9	150	900	µg/kg
Benzo(a)pyrene	50-32-8	4.6	1.37	4.6	15	90	µg/kg
Benzo(b)fluoranthene	205-99-2	4.8	1.43	4.8	150	900	µg/kg
Benzo(g,h,i)perylene	191-24-2	4.8	1.43	4.8	NS	NS	µg/kg
Benzo(k)fluoranthene	207-08-9	5.0	1.48	5.0	1,500	9,000	µg/kg
Bis(2-chloroethoxy)methane	111-91-1	100	20	67	180,000	NS	µg/kg
Bis(2-chloroethyl)ether	111-44-4	100	14	46	190	660	µg/kg
Bis(2-ethylhexyl)phthalate	117-81-7	100	28	93	35,000	46,000	µg/kg
Butylbenzylphthalate	85-68-7	100	24	80	12,000,000	930,000	µg/kg
Carbazole	86-74-8	100	11	36	24,000	600	µg/kg
Chrysene	218-01-9	5.2	1.53	5.2	1,500	88,000	µg/kg
Di-n-butylphthalate	84-74-2	140	40	140	NS	2,300,000	µg/kg
Di-n-octylphthalate	117-84-0	130	39	130	NS	1,600,000	µg/kg
Dibenzo(a,h)anthracene	53-70-3	4.87	1.46	4.87	6,215	90	µg/kg
Dibenzofuran	132-64-9	130	37	130	NS	NS	µg/kg
Diethylphthalate	84-66-2	130	38	130	49,000,000	470,000	µg/kg
Dimethylphthalate	131-11-3	100	26	86	NS	NS	µg/kg
Fluoranthene	206-44-0	5.3	1.56	5.3	2,300,000	3,100,000	µg/kg
Fluorene	86-73-7	5.2	1.56	5.2	2,300,000	560,000	µg/kg
Hexachlorobenzene	118-74-1	100	15	50	300	400	µg/kg
Hexachlorobutadiene	87-68-3	110	31	110	6,200	NS	µg/kg
Hexachlorocyclopentadiene	77-47-4	100	26	86	370,000	10,000	µg/kg
Hexachloroethane	67-72-1	100	17	55	350,000	500	µg/kg
Indeno(1,2,3-cd)pyrene	193-39-5	4.6	1.38	4.6	150	900	µg/kg
Isophorone	78-59-1	100	27	91	510,000	8,000	µg/kg
N-Nitroso-di-n-propylamine	621-64-7	110	30	110	**69**	**1.80**	µg/kg
N-Nitrosodimethylamine	62-75-9	460	140	460	99,000	NS	µg/kg
Naphthalene	91-20-3	4.4	1.33	4.4	3,900	12,000	µg/kg
Nitrobenzene	98-95-3	140	40	140	31,000	**260**	µg/kg
Pentachlorophenol	87-86-5	500	70	240	3,000	**30**	µg/kg
Phenanthrene	85-01-8	5.3	1.58	5.3	NS	NS	µg/kg
Phenol	108-95-2	410	130	410	18,000,000	100,000	µg/kg
Pyrene	129-00-0	5.0	1.48	5.0	1,700,000	2,300,000	µg/kg

REFERENCE LIMITS AND EVALUTATIONS TABLE
SOLIDS
SOIL & SEDIMENT

SVOC	Surrogates	CAS #	Lower	Upper	Units
	Surr: 2,4,6-Tribromophenol	118-79-6	35	125	% Recovery
	Surr: 2-Fluorobiphenyl	321-60-8	45	105	% Recovery
	Surr: 2-Fluorophenol	367-12-4	35	105	% Recovery
	Surr: Nitrobenzene-d5	4165-60-0	35	100	% Recovery
	Surr: o-terphenyl-d14	5142-67-6	30	125	% Recovery
	Surr: Phenol-d5	4165-62-2	40	100	% Recovery
	Surr: Terphenyl-d14	98904-43-9	30	125	% Recovery

METALS BY ICP QSM
SW 846 METHOD 6010B
Preparatory SW 846 Method 3050B

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	CAS #	RL	MDL	PQL	RSL	TACO	Units
					Residential Soil		
Aluminum	7429-90-5	0.22	0.07	0.22	77,000	NS	mg/kg
Antimony	7440-36-0	0.18	0.05	0.18	31	6	mg/kg
Arsenic	7440-38-2	0.28	0.08	0.28	0.39	50	mg/kg
Barium	7440-39-3	0.055	0.017	0.055	15,000	2,000	mg/kg
Beryllium	7440-41-7	0.017	0.005	0.017	160	4	mg/kg
Cadmium	7440-43-9	0.014	0.004	0.014	70	5	mg/kg
Calcium	7440-70-2	1.1	0.3	1.1	NS	NS	mg/kg
Chromium	7440-47-3	0.043	0.013	0.043	230	100	mg/kg
Cobalt	7440-48-4	0.030	0.009	0.030	NS	1,000	mg/kg
Copper	7440-50-8	0.19	0.06	0.19	3,100	650	mg/kg
Cyanide[a]	57-12-5	14	4	14	1,600,000	200	mg/kg
Iron	7439-89-6	1.2	0.4	1.2	55,000	5,000	mg/kg
Lead	7439-92-1	0.11	0.03	0.11	400	7.5	mg/kg
Magnesium	7439-95-4	0.34	0.10	0.34	NS	325000	mg/kg
Manganese	7439-96-5	0.11	0.03	0.11	1,800	150	mg/kg
Mercury[b]	7439-97-6	0.0038	0.0012	0.0038	6.7	2	mg/kg
Nickel	7440-02-0	0.045	0.014	0.045	1,600	100	mg/kg
Potassium	7440-09-7	13	4	13	NS	NS	mg/kg
Selenium	7782-49-2	0.24	0.07	0.24	390	50	mg/kg
Silver	7440-22-4	0.074	0.022	0.074	390	50	mg/kg
Sodium	7440-23-5	7.2	2.2	7.2	NS	NS	mg/kg
Thallium	7440-28-0	0.18	0.05	0.18	5.1	2	mg/kg
Vanadium	7440-62-2	0.057	0.018	0.057	550	49	mg/kg
Zinc	7440-66-6	0.11	0.03	0.11	23,000	5,000	mg/kg

POLYCHLORINATED BIPHENYLS (PCBs) BY GC
SW 846 METHOD 8082A
Preparatory SW 846 Method 3545

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	CAS #	RL	MDL	PQL	RSL	TACO	Units
					Residential Soil		
Aroclor-1016	12674-11-2	31.0	9.0	31.0	3,900	NS	µg/kg
Aroclor-1221	11104-28-2	41.0	12.0	41.0	170	NS	µg/kg
Aroclor-1232	11141-16-5	47.0	14.0	47.0	170	NS	µg/kg
Aroclor-1242	53469-21-9	34.0	10.0	34.0	220	NS	µg/kg
Aroclor-1248	12672-29-6	30.0	9.0	30.0	220	NS	µg/kg
Aroclor-1254	11097-69-1	30.0	3.0	30.0	220	NS	µg/kg
Aroclor-1260	11096-82-5	30.0	6.0	30.0	220	NS	µg/kg
Aroclor-1262	37324-23-5	30.0	6.0	30.0	NS	NS	µg/kg
Aroclor-1268	11100-14-4	30.0	5.0	30.0	NS	NS	µg/kg

Surrogates		Lower	Upper	Units
Surr: 2,4,5,6-TCMX	877-09-8	50	150	% Recovery
Surr: DCBP	2051-24-3	60	125	% Recovery

REFERENCE LIMITS AND EVALUTATIONS TABLE
SOLIDS
SOIL & SEDIMENT

ORGANOCHLORINE PESTICIDES BY GC
SW 846 METHOD 8081A
Preparatory SW 846 Method 3545

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**TACO**	**Units**
					Residential Soil		
4,4'-DDD	72-54-8	1.20	0.30	1.20	2,000	3,000	µg/kg
4,4'-DDE	72-55-9	1.10	0.30	1.10	1,400	2,000	µg/kg
4,4'-DDT	50-29-3	1.50	0.50	1.50	1,700	2,000	µg/kg
Aldrin	309-00-2	1.70	0.50	1.70	29	940	µg/kg
alpha-BHC	319-84-6	1.90	0.60	1.90	77	7.4	µg/kg
alpha-Chlordane	5103-71-9	1.10	0.30	1.10	1,600	NS	µg/kg
beta-BHC	319-85-7	2.00	0.60	2.00	270	NS	µg/kg
delta-BHC	319-86-8	1.10	0.30	1.10	NS	NS	µg/kg
Dieldrin	60-57-1	1.20	0.30	1.20	NS	603	µg/kg
Endosulfan I	959-98-8	2.20	0.70	2.20	370,000	18,000	µg/kg
Endosulfan II	33213-65-9	1.20	0.30	1.20	370,000	18,000	µg/kg
Endosulfan sulfate	1031-07-8	3.10	0.90	3.10	370,000	18,000	µg/kg
Endrin	72-20-8	1.40	0.40	1.40	18,000	1,000	µg/kg
Endrin aldehyde	7421-93-4	3.60	1.10	3.60	18,000	1,000	µg/kg
Endrin ketone	53494-70-5	2.80	0.80	2.80	18,000	1,000	µg/kg
gamma-Chlordane	5103-74-2	1.10	0.30	1.10	520	NS	µg/kg
Heptachlor	76-44-8	1.20	0.40	1.20	110	871	µg/kg
Heptachlor epoxide	1024-57-3	1.70	0.50	1.70	53	1,005	µg/kg
Lindane	58-89-9	1.60	0.50	1.60	520	9	µg/kg
Methoxychlor	72-43-5	2.30	0.70	2.30	310,000	160,000	µg/kg
Toxaphene	8001-35-2	30.0	5.00	30.0	440	600	µg/kg
Surrogates			**Lower**	**Upper**			**Units**
SURR:2,4,5,6-CL4-m-xylene	877-09-8		70	125			% Recovery
SURR:Decachlorobiphenyl	2051-24-3		55	130			% Recovery

NITROAROMATICS AND NITRAMINES BY HPLC
SW 846 METHOD 8330A
Preparatory SW 846 Method 3535A

		LABORATORY LIMITS			DATA QUALITY LIMITS		
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**TACO**	**Units**
					Residential Soil		
1,3,5-Trinitrobenzene	99-35-4	0.4	0.06	0.20	1,800	NS	mg/kg
2,4,6-Trinitrotoluene	118-96-7	0.4	0.06	0.20	16	NS	mg/kg
2,4-Dinitrotoluene	121-14-2	0.4	0.07	0.30	120	250	mg/kg
2,6-Dinitrotoluene	606-20-2	0.4	0.07	0.30	61	260	mg/kg
2-Amino-4,6-dinitrotoluene	35572-78-2	0.4	0.07	0.30	120	NS	mg/kg
2-Nitrotoluene	88-72-2	0.4	0.07	0.30	780	NS	mg/kg
3-Nitrotoluene	99-08-1	0.4	0.08	0.30	NS	NS	mg/kg
4-Amino-2,6-dinitrotoluene	19406-51-0	0.4	0.10	0.40	120	NS	mg/kg
4-Nitrotoluene	99-99-0	0.4	0.07	0.30	30	NS	mg/kg
HMX	2691-41-0	0.4	0.10	0.40	3,100	NS	mg/kg
Nitrobenzene	98-95-3	0.4	0.07	0.30	31	100	mg/kg
Nitrocellulose[a]	9004-70-0	5	5	16	NS	NS	mg/kg
Nitroglycerin	55-63-0	3.40	1.10	3.40	6.1	NS	mg/kg
Nitroguanidine	556-88-7	0.14	0.05	0.14	6,100	NS	mg/kg
PETN	78-11-5	6.40	2.00	6.40	NS	NS	mg/kg
RDX	121-82-4	0.4	0.12	0.40	4.4	NS	mg/kg
Tetryl	479-45-8	0.4	0.11	0.40	240	NS	mg/kg
Surrogates			**Lower**	**Upper**			**Units**
1,2-Dinitrobenzene	528-29-0		50	150			% Recovery

LEGEND FOR SOLIDS TABLES

CAS #	Chemical abstract number
RL	Reporting Limit
MDL	Method Detection Limit
PQL	Practical Quantitation Limit
RSL	USEPA Sept 2008 Regional Screening Level for Residential Soil
TACO	Illinois Tier 1 Soil Remediation Objectives for Residential Properties
NS	None Specified
mg/kg	Milligrams per kilogram
µg/kg	Micrograms per kilogram
[a]	Total and amenable cyanide determination by SW 846 Method 9012A
[b]	Mercury determination by SW 846 Method 7471A
VALUE	Compounds for which Data Quanitation Limit is lower than RL are shaded
VALUE	Compounds for which Data Quanitation Limit is lower than MDL are shaded and bolded

REFERENCE LIMITS AND EVALUATIONS TABLE
AQUEOUS MATRICES
GROUNDWATER & SURFACE WATER

VOLATILE ORGANIC COMPOUNDS BY GC/MS (LOW LEVEL)
SW 846 METHOD 8260B
Preparatory SW 846 Method 5035

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
1,1,1-Trichloroethane	71-55-6	0.2	0.05	0.17	9,100	390	NS	200	µg/L
1,1,2,2-Tetrachloroethane	79-34-5	0.2	0.019	0.064	0.067	140	3.2	NS	µg/L
1,1,2-Trichloroethane	79-00-5	0.2	0.06	0.20	0.24	4400	12	5	µg/L
1,1-Dichloroethane	75-34-3	0.2	0.06	0.19	2.4	2000	NS	700	µg/L
1,1-Dichloroethene	75-35-4	0.2	0.05	0.16	340	240	0.95	7	µg/L
1,2,3-Trichlorobenzene	87-61-6	0.22	0.07	0.22	NS	2.8	NS	NS	µg/L
1,2,4-Trichlorobenzene	120-82-1	0.2	0.06	0.19	180,000	72	NS	70	µg/L
1,2-Dibromo-3-chloropropane	96-12-8	0.2	0.05	0.17	**0.0003**	190	NS	0.2	µg/L
1,2-Dichlorobenzene	95-50-1	0.2	0.05	0.17	370	170	NS	NS	µg/L
1,2-Dichloroethane	107-06-2	0.2	0.05	0.16	0.15	4500	23	5	µg/L
1,2-Dichloropropane	78-87-5	0.2	0.05	0.17	0.39	380	5.7	5	µg/L
1,3-Dichlorobenzene	541-73-1	0.2	0.027	0.089	NS	200	NS	NS	µg/L
1,3-Dichloropropane	142-28-9	0.25	0.07	0.25	730	NS	NS	NS	µg/L
1,4-Dichlorobenzene	106-46-7	0.2	0.04	0.13	0.43	620	NS	NS	µg/L
112Trichloro122trifluoroethane	76-13-1	0.2	0.027	0.090	59,000	NS	NS	NS	µg/L
2-Butanone	78-93-3	2.0	0.6	1.9	7,100	26000	NS	4200	µg/L
2-Hexanone	591-78-6	5.2	1.6	5.2	NS	950	NS	NS	µg/L
4-Methyl-2-pentanone	108-10-1	2.8	0.8	2.8	2,000	1400	NS	NS	µg/L
Acetone	67-64-1	5.2	1.5	5.2	22,000	120000000	NS	6300	µg/L
Benzene	71-43-2	0.2	0.05	0.17	0.41	860[e]	NS	3	µg/L
Bromochloromethane	74-97-5	0.2	0.028	0.095	NS	NS	NS	NS	µg/L
Bromodichloromethane	75-27-4	0.2	0.03	0.12	1.1	1	13	NS	µg/L
Bromoform	75-25-2	0.2	0.04	0.13	8.5	NS	50	NS	µg/L
Bromomethane	74-83-9	0.25	0.07	0.25	8.7	NS	NS	NS	µg/L
Carbon disulfide	75-15-0	0.31	0.09	0.31	1,000	20	NS	700	µg/L
Carbon tetrachloride	56-23-5	0.2	0.022	0.074	0.2	280	1.4	5	µg/L
Chlorobenzene	108-90-7	0.2	0.04	0.15	91	79	4500	NS	µg/L
Chloroethane	75-00-3	0.23	0.07	0.23	21,000	1000	NS	NS	µg/L
Chloroform	67-66-3	0.2	0.022	0.073	0.19	150	130	0.2	µg/L
Chloromethane	74-87-3	0.2	0.05	0.17	1.8	1300	NS	NS	µg/L
cis-1,2-Dichloroethene	156-59-2	0.2	0.05	0.17	370	NS	NS	70	µg/L
cis-1,3-Dichloropropene	10061-01-5	0.2	0.017	0.056	0.43	NS	NS	NS	µg/L
Dibromochloromethane	124-48-1	0.2	0.026	0.085	0.8	NS	9.8	NS	µg/L
Ethylbenzene	100-41-4	0.2	0.024	0.079	1.5	14[e]	NS	NS	µg/L
Isopropylbenzene	98-82-8	0.2	0.04	0.12	NS	NS	NS	700	µg/L
m & p-Xylene	1330-20-7	0.26	0.08	0.26	2,900	360[e]	NS	10000	µg/L
Methyl tert-butyl ether	1634-04-4	0.26	0.08	0.26	12	5400	NS	70	µg/L
Methylene chloride	75-09-2	0.59	0.18	0.59	4.8	1400	330	NS	µg/L
o-Xylene	95-47-6	0.2	0.023	0.075	1,400	NS	NS	NS	µg/L
Styrene	100-42-5	0.2	0.022	0.075	1,600	200	NS	100	µg/L
tert-Butylbenzene	98-06-6	0.2	0.021	0.069	NS	7.7	NS	NS	µg/L
Tetrachloroethene	127-18-4	0.2	0.05	0.16	**0.11**	150	NS	5	µg/L
Toluene	108-88-3	0.2	0.06	0.19	2,300	600[e]	NS	1000	µg/L
trans-1,2-Dichloroethene	156-60-5	0.21	0.06	0.21	110	NS	34000	100	µg/L
trans-1,3-Dichloropropene	10061-02-6	0.2	0.017	0.058	0.43	NS	NS	NS	µg/L
Trichloroethene	79-01-6	0.2	0.05	0.16	1.7	940	2.5	5	µg/L
Trichlorofluoromethane	75-69-4	0.2	0.06	0.20	1,300	NS	250000	2100	µg/L
Vinyl chloride	75-01-4	0.2	0.013	0.044	**0.016**	930	0.25	2	µg/L

Surrogates		**Lower**	**Upper**	**Units**
Bromofluorobenzene	460-00-4	75	120	% Recovery
1,2 Dichloroethane-d4	17060-07-0	70	120	% Recovery
d8-Toluene	2037-26-5	85	120	% Recovery
Dibromofluoromethane	1868-53-7	85	115	% Recovery

REFERENCE LIMITS AND EVALUATIONS TABLE
AQUEOUS MATRICES
GROUNDWATER & SURFACE WATER

SEMIVOLATILE ORGANIC COMPOUNDS (LOW LEVEL) + PAH SIM QSM
SW 846 METHOD 8270C
Preparatory SW 846 Method 3540C

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
2,4,5-Trichlorophenol	95-95-4	2.65	0.79	2.65	3,700	NS	3.2	NS	µg/L
2,4,6-Trichlorophenol	88-06-2	2.86	0.86	2.86	6.1	6.3	2.1	NS	µg/L
2,4-Dichlorophenol	120-83-2	2.03	0.61	2.03	110	83	NS	NS	µg/L
2,4-Dimethylphenol	105-67-9	3.96	1.19	3.96	730	220	NS	NS	µg/L
2,4-Dinitrophenol	51-28-5	5.08	1.52	5.05	73	4.1	NS	NS	µg/L
2,4-Dinitrotoluene	121-14-2	0.62	0.19	1	73	320	2.6	**0.02**	µg/L
2,6-Dinitrotoluene	606-20-2	0.38	0.12	1	37	150	0.4	**0.31**	µg/L
2-Chloronaphthalene	91-58-7	0.34	0.10	1	2,900	30	NS	NS	µg/L
2-Chlorophenol	95-57-8	2.27	0.68	2.27	180	41	NS	NS	µg/L
2-Methylnaphthalene	91-57-6	0.020	0.005	0.02	150	NS	NS	2.8	µg/L
2-Methylphenol	95-48-7	2.88	0.87	2.88	1,800	370.00	NS	350	µg/L
2-Nitroaniline	88-74-4	0.35	0.11	1	NS	200	NS	NS	µg/L
2-Nitrophenol	88-75-5	2.01	0.60	2.01	NS	540	NS	NS	µg/L
3 & 4-Methylphenol	1319-77-3	2.23	0.67	2.23	NS	NS	NS	NS	µg/L
3,3'-Dichlorobenzidine	91-94-1	9.86	2.96	9.86	NS	NS	NS	NS	µg/L
3-Nitroaniline	99-09-2	0.70	0.21	1	NS	NS	NS	NS	µg/L
4,6-Dinitro-2-methylphenol	534-52-1	3.08	0.93	3.08	NS	**2.3**	NS	NS	µg/L
4-Bromophenyl-phenyl ether	101-55-3	0.51	0.15	1	150	2.9	NS	NS	µg/L
4-Chloro-3-methylphenol	59-50-7	3.71	1.11	3.71	NS	25	NS	NS	µg/L
4-Chloroaniline	106-47-8	0.57	0.17	1	150	0.2	NS	NS	µg/L
4-Chlorophenyl-phenyl ether	7005-72-3	0.25	0.08	1	NS	NS	NS	NS	µg/L
4-Nitroaniline	100-01-6	1.53	0.46	1.53	3.2	120	NS	NS	µg/L
4-Nitrophenol	100-02-7	2.07	0.62	2.07	NS	1900	NS	NS	µg/L
Acenaphthene	83-32-9	0.02	0.005	0.02	2,200	62	NS	420	µg/L
Acenaphthylene	208-96-8	0.020	0.004	0.02	NS	15	NS	NS	µg/L
Anthracene	120-12-7	0.020	0.004	0.02	11,000	0.53	35	43.4	µg/L
Benzo(a)anthracene	56-55-3	0.020	0.004	0.02	0.029	NS	0.16	0.13	µg/L
Benzo(a)pyrene	50-32-8	0.022	0.007	0.022	**0.0029**	NS	**0.016**	0.2	µg/L
Benzo(b)fluoranthene	205-99-2	0.021	0.006	0.021	0.029	NS	0.16	0.18	µg/L
Benzo(g,h,i)perylene	191-24-2	0.022	0.007	0.022	NS	NS	NS	NS	µg/L
Benzo(k)fluoranthene	207-08-9	0.020	0.006	0.02	0.29	NS	1.6	0.8	µg/L
Bis(2-chloroethoxy)methane	111-91-1	0.29	0.09	1	110	NS	NS	NS	µg/L
Bis(2-chloroethyl)ether	111-44-4	0.66	0.20	1	**0.012**	1.9	**0.42**	NS	µg/L
Bis(2-ethylhexyl)phthalate	117-81-7	2.03	0.61	2.03	4.8	17	2.8	NS	µg/L
Butylbenzylphthalate	85-68-7	0.38	0.11	1	7,300	23	1.7	NS	µg/L
Carbazole	86-74-8	0.45	0.14	1	3.4	7.4	NS	NS	µg/L
Chrysene	218-01-9	0.020	0.004	0.02	2.9	NS	16	1.6	µg/L
Di-n-butylphthalate	84-74-2	2.51	0.75	2.51	NS	48	NS	700	µg/L
Di-n-octylphthalate	117-84-0	0.52	0.16	1	NS	NS	NS	20	µg/L
Dibenzo(a,h)anthracene	53-70-3	0.020	0.005	0.02	**0.0029**	NS	0.016	0.3	µg/L
Dibenzofuran	132-64-9	0.28	0.08	1	NS	15	NS	NS	µg/L
Diethylphthalate	84-66-2	0.56	0.17	1	29,000	250	NS	5600	µg/L
Dimethylphthalate	131-11-3	0.30	0.09	1	NS	260	NS	NS	µg/L
Fluoranthene	206-44-0	0.020	0.005	0.02	1,500	1.8	120	206	µg/L
Fluorene	86-73-7	0.020	0.004	0.02	1,500	16	5	280	µg/L
Hexachlorobenzene	118-74-1	0.74	0.22	1	**0.042**	NS	0.25	NS	µg/L
Hexachlorobutadiene	87-68-3	0.99	0.30	1	**0.86**	2.8	NS	NS	µg/L
Hexachlorocyclopentadiene	77-47-4	0.41	0.12	1	220	**0.1**	NS	50	µg/L
Hexachloroethane	67-72-1	0.24	0.07	1	4.8	31	2.9	NS	µg/L
Indeno(1,2,3-cd)pyrene	193-39-5	0.021	0.006	0.021	0.029	NS	0.16	0.43	µg/L
Isophorone	78-59-1	0.38	0.11	1	71	960	0.76	NS	µg/L
N-Nitroso-di-n-propylamine	621-64-7	0.60	0.18	1	**0.0096**	NS	NS	NS	µg/L
N-Nitrosodiphenylamine & Diphn	86-30-6/122-39-4	0.64	0.19	2	14	NS	NS	NS	µg/L
Naphthalene	91-20-3	0.021	0.006	0.021	0.14	68	NS	140	µg/L
Pentachlorophenol	87-86-5	2.95	0.89	2.95	**0.56**	13	**2.5**	**1**	µg/L
Phenanthrene	85-01-8	0.020	0.004	0.020	NS	3.7	NS	NS	µg/L
Phenol	108-95-2	1.02	0.31	0.31	11,000	NS	NS	100	µg/L
Pyrene	129-00-0	0.020	0.004	0.020	1,100	NS	4	135	µg/L

REFERENCE LIMITS AND EVALUATIONS TABLE
AQUEOUS MATRICES
GROUNDWATER & SURFACE WATER

SVOCs	Surrogates		Lower	Upper	Units
	Surr: 2,4,6-Tribromophenol	118-79-6	50	150	% Recovery
	Surr: 2-Fluorobiphenyl	321-60-8	50	150	% Recovery
	Surr: 2-Fluorophenol	367-12-4	50	150	% Recovery
	Surr: Nitrobenzene-d5	4165-60-0	50	150	% Recovery
	Surr: o-terphenyl-d14	5142-67-6	50	150	% Recovery
	Surr: Phenol-d5	4165-62-2	50	150	% Recovery
	Surr: Terphenyl-d14	98904-43-9	50	150	% Recovery

METALS BY ICP-MS QSM
SW 846 METHOD 6010B
Preparatory SW 846 Method 3050B

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	CAS #	RL	MDL	PQL	RSL	SW WQ		GW STD	Units
					Tap Water	Aquatic	Human	Class I	
Aluminum	7429-90-5	24	7	24	37,000	190[e]	NS	NS	µg/L
Antimony	7440-36-0	11	3	11	15	320	NS	**6**	µg/L
Arsenic[a]	7440-38-2	2.6	0.8	2.6	**0.045**	NS	NS	10	µg/L
Barium	7440-39-3	1.6	0.5	1.6	7,300	NS	NS	2000	µg/L
Beryllium	7440-41-7	0.41	0.12	0.41	73	NS	NS	4	µg/L
Cadmium	7440-43-9	0.53	0.16	0.53	18	1.03[e]	NS	5	µg/L
Calcium	7440-70-2	29	9	29	NS	NS	NS	NS	µg/L
Chromium	7440-47-3	3.0	1.0	3.0	110	178[e]	NS	100	µg/L
Cobalt	7440-48-4	1.1	0.3	1.1	NS	NS	NS	1000	µg/L
Copper	7440-50-8	6.3	1.9	6.3	1,500	11.4[e]	NS	650	µg/L
Cyanide[b]	57-12-5	21	6	21	730	5.2[e]	NS	200	µg/L
Iron	7439-89-6	25	8	25	26,000	NS	NS	5000	µg/L
Lead	7439-92-1	4.9	1.5	4.9	NS	15.9[e]	NS	7.5	µg/L
Magnesium	7439-95-4	22	7	22	NS	NS	NS	NS	µg/L
Manganese	7439-96-5	6.5	1.9	6.5	880	NS	NS	150	µg/L
Mercury[c]	7439-97-6	0.13	0.04	0.13	0.63	1.1[e]	0.012[f]	2	µg/L
Nickel	7440-02-0	3.5	1.0	3.5	730	5.0[e]	NS	100	µg/L
Potassium	7440-09-7	470	140	140	NS	NS	NS	NS	µg/L
Selenium	7782-49-2	7.7	2.3	7.7	180	NS	NS	50	µg/L
Silver	7440-22-4	3.2	1.0	3.2	180	NS	NS	50	µg/L
Sodium	7440-23-5	640	190	640	NS	NS	NS	NS	µg/L
Thallium[d]	7440-28-0	1.4	0.4	1.4	2.4	11	1.2	2	µg/L
Vanadium	7440-62-2	3.0	0.9	3.0	260	NS	NS	49	µg/L
Zinc	7440-66-6	1.6	0.5	1.6	11,000	21.6[e]	NS	5000	µg/L

POLYCHLORINATED BIPHENYLS (PCBs) QCM/LCG
SW 846 METHOD 8082A
Preparatory SW846 Method 3510

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	CAS #	RL	MDL	PQL	RSL	SW WQ		GW STD	Units
					Tap Water	Aquatic	Human	Class I*	
Aroclor-1016	12674-11-2	0.379	0.114	0.379	0.96	NS	15	0.5	µg/L
Aroclor-1221	11104-28-2	0.300	0.071	0.300	**0.0068**	NS	15	0.5	µg/L
Aroclor-1232	11141-16-5	0.468	0.140	0.468	**0.0068**	NS	15	0.5	µg/L
Aroclor-1242	53469-21-9	0.322	0.096	0.322	**0.034**	NS	15	0.5	µg/L
Aroclor-1248	12672-29-6	0.393	0.118	0.393	**0.034**	NS	15	0.5	µg/L
Aroclor-1254	11097-69-1	0.300	0.076	0.300	**0.034**	NS	15	0.5	µg/L
Aroclor-1260	11096-82-5	0.384	0.115	0.384	**0.034**	NS	15	0.5	µg/L
Aroclor-1262	37324-23-5	0.468	0.140	0.468	NS	NS	15	0.5	µg/L
Aroclor-1268	11100-14-4	0.300	0.035	0.300	NS	NS	15	0.5	µg/L

Surrogates		Lower	Upper	Units
Surr: 2,4,5,6-TCMX	877-09-8	50	150	% Recovery
Surr: DCBP	2051-24-3	30	145	% Recovery

REFERENCE LIMITS AND EVALUATIONS TABLE
AQUEOUS MATRICES
GROUNDWATER & SURFACE WATER

ORGANOCHLORINE PESTICIDES BY GC
SW 846 METHOD 8081A
Preparatory SW846 Method 3510

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
4,4'-DDD	72-54-8	0.020	0.006	0.020	0.28	NS	**0.00027**	NS	µg/L
4,4'-DDE	72-55-9	0.020	0.006	0.020	0.2	NS	**0.00019**	NS	µg/L
4,4'-DDT	50-29-3	0.022	0.007	0.022	0.2	0.000001	**0.00019**	NS	µg/L
Aldrin	309-00-2	0.017	0.006	0.017	**0.004**	NS	46	NS	µg/L
alpha-BHC	319-84-6	0.016	0.005	0.016	0.011	2.5	**0.0042**	NS	µg/L
alpha-Chlordane	5103-71-9	0.030	0.009	0.030	0.19	NS	NS	NS	µg/L
beta-BHC	319-85-7	0.030	0.009	0.030	0.037	NS	**0.015**	NS	µg/L
delta-BHC	319-86-8	0.017	0.005	0.017	NS	NS	0.0414	NS	µg/L
Dieldrin	60-57-1	0.018	0.006	0.018	**0.0042**	0.056	**0.000046**	NS	µg/L
Endosulfan I	959-98-8	0.028	0.009	0.028	220	**0.0056**	NS	NS	µg/L
Endosulfan II	33213-65-9	0.022	0.007	0.022	220	**0.0056**	NS	NS	µg/L
Endosulfan sulfate	1031-07-8	0.020	0.006	0.020	220	**0.0056**	NS	NS	µg/L
Endrin	72-20-8	0.020	0.006	0.020	11	0.033	NS	2	µg/L
Endrin aldehyde	7421-93-4	0.028	0.009	0.028	11	NS	NS	NS	µg/L
Endrin ketone	53494-70-5	0.021	0.007	0.021	11	NS	NS	NS	µg/L
gamma-Chlordane	5103-74-2	0.024	0.007	0.024	0.19	NS	NS	NS	µg/L
Heptachlor	76-44-8	0.019	0.006	0.019	**0.015**	0.0038	68	0.4	µg/L
Heptachlor epoxide	1024-57-3	0.021	0.007	0.021	**0.0074**	**0.0038**	36	0.2	µg/L
Lindane	58-89-9	0.022	0.007	0.022	0.061	NS	**0.02**	NS	µg/L
Methoxychlor	72-43-5	0.020	0.006	0.020	180	0.02	NS	40	µg/L
Toxaphene	8001-35-2	0.562	0.176	0.562	0.6	**0.0002**	**0.00024**	3	µg/L
Surrogates			**Lower**	**Upper**					**Units**
SURR:2,4,5,6-CL4-m-xylene	877-09-8		25	140					% Recovery
SURR:Decachlorobiphenyl	2051-24-3		30	135					% Recovery

NITROAROMATICS AND NITRAMINES BY HPLC
SW 846 METHOD 8330A[a]
Preparatory SW 846 Method 3535A

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
1,3,5-Trinitrobenzene	99-35-4	1.0	0.30	0.80	1,100	15	**0.58**	840	µg/L
2,4,6-Trinitrotoluene	118-96-7	1.0	0.30	1.00	2.2	NS	NS	14	µg/L
2,4-Dinitrotoluene	121-14-2	1.0	0.30	1.00	73	320	2.6	**0.02**	µg/L
2,6-Dinitrotoluene	606-20-2	1.0	0.30	0.90	37	150	**0.4**	**0.31**	µg/L
2-Amino-4,6-dinitrotoluene	35572-78-2	1.0	0.30	0.80	73	7.9	NS	NS	µg/L
2-Nitrotoluene	88-72-2	1.0	0.30	1.00	370	62	NS	NS	µg/L
3-Nitrotoluene	99-08-1	1.0	0.30	1.00	NS	NS	NS	NS	µg/L
4-Amino-2,6-dinitrotoluene	19406-51-0	1.0	0.30	0.90	73	14	NS	NS	µg/L
4-Nitrotoluene	99-99-0	1.0	0.30	0.90	4.2	NS	NS	NS	µg/L
HMX	2691-41-0	1.0	0.30	0.90	1800	260	NS	1400	µg/L
Nitrobenzene	98-95-3	1.0	0.30	0.90	3.4	8500	530	3.5	µg/L
Nitrocellulose	9004-70-0	1.0	1.0	3.0	NS	NS	NS	NS	mg/L
Nitroglycerin	55-63-0	10	2.30	7.70	**3.7**	NS	NS	NS	µg/L
Nitroguanidine	556-88-7	84	26	84	3,700	NS	NS	NS	µg/L
PETN	78-11-5	20	4.40	14.50	NS	NS	NS	NS	µg/L
RDX	121-82-4	1.0	0.30	0.70	0.61	500	NS	84	µg/L
Tetryl	479-45-8	1.0	0.20	0.70	150	NS	NS	NS	µg/L
Surrogates			**Lower**	**Upper**					**Units**
1,2-Dinitrobenzene	528-29-0		50	150					% Recovery

TOTAL ORGANIC CARBON
SW 846 METHOD 9060A
Preparatory Method Lloyd Kahn

					RSL	SW WQ			
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**Tap Water**	**Aquatic**	**Human**	**GW STD**	**Units**
Total Organic Carbon	NA	0.8	0.8	2.8	NS	NS	NS	NS	mg/L

REFERENCE LIMITS AND EVALUATIONS TABLE
AQUEOUS MATRICES
GROUNDWATER & SURFACE WATER

TOTAL ORGANIC HALIDES (TOX)
SW 846 METHOD 9020B

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
TOX	NA	20	11	NS	NS	NS	NS	NS	µg/L

DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY
SW 846 METHOD 9056A
Preparatory SW 846 Method 353.2

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
Nitrite	14797-65-0	0.42	0.12	0.42	3.7	NS	NS	1	mg/L
Nitrate	14797-55-8	0.42	0.12	0.42	58	NS	NS	10	mg/L

DETERMINATION OF PERCHLORATE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY COUPLED WITH ELECTROSPRAY IONIZATION TANDEM MASS SPECTROMETRY
SW 846 METHOD 6850

		LABORATORY LIMITS			DATA QUALITY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**RSL**	**SW WQ**		**GW STD**	**Units**
					Tap Water	**Aquatic**	**Human**	**Class I**	
Perchlorate	14797-73-0	0.05	0.01	0.05	26	NS	NS	NS	µg/L

LEGEND FOR AQEUOUS TABLES

CAS # Chemical abstract number
RL Reporting Limit
MDL Method Detection Limit
PQL Practical Quantitation Limit
RSL USEPA Sept 2008 Regional Screening Level for Tapwater
GW STD Illinois Groundwater Quality Standards for Class I: Potable Resource Groundwater
SW WQ Illinois Surface Water Quality Standard or Illionois Chronic Aquatic Life and Human Health WQ Criteria
NS None Specified
mg/L Milligrams per liter
µg/L Micrograms per liter
[a] Arsenic determination by SW 846 Method 7060A
[b] Cyanide determination by SW 846 Method 9012A
[c] Mercury determination by SW 846 Method 7471A
[d] Thallium determination by SW 846 Method 7841
[e] IEPA Aquatic Life Standards for dissolved metals, total cyanide, and VOCs from Title 35, Subtitle C, Chapter I, Part 302.
[f] IEPA Human Health Standard for total mercury from Title 35, Subtitle C, Chapter I, Part 302.
[*] Value for Total PCBs
VALUE Compounds for which Data Quanitation Limit is lower than RL are shaded
VALUE Compounds for which Data Quanitation Limit is lower than MDL are shaded and bolded

REFERENCE LIMITS AND EVALUATIONS TABLE
Investigative Derived Waste (IDW)

TCLP VOLATILE ORGANIC COMPOUNDS
SW 846 METHOD 1311 / 8260B

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
1,1-Dichloroethene	75-35-4	0.7	0.0004	0.0013	0.7	mg/L
1,2-Dichloroethane	107-06-2	0.5	0.0003	0.0011	0.5	mg/L
2-Butanone	78-93-3	200	0.004	0.014	200	mg/L
Benzene	71-43-2	0.5	0.00016	0.00055	0.5	mg/L
Carbon tetrachloride	56-23-5	0.5	0.0004	0.0013	0.5	mg/L
Chlorobenzene	108-90-7	100	0.0003	0.0011	100	mg/L
Chloroform	67-66-3	6	0.00022	0.00072	6	mg/L
Tetrachloroethene	127-18-4	0.7	0.0004	0.0013	0.7	mg/L
Trichloroethene	79-01-6	0.5	0.00015	0.00048	0.5	mg/L
Vinyl chloride	75-01-4	0.2	0.00015	0.00049	0.2	mg/L
Surrogates			**Lower**	**Upper**		**Units**
1,2 Dichloroethane-d4	17060-07-0		66	132		% Recovery
Bromofluorobenzene	460-00-4		64	152		% Recovery
d8-Toluene	2037-26-5		71	137		% Recovery
Dibromofluoromethane	1868-53-7		69	145		% Recovery

TCLP SEMIVOLATILE ORGANIC COMPOUNDS
SW 846 METHOD 1311 / 8270C

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
1,4-Dichlorobenzene	106-46-7	7.5	0.00008	0.00028	7.5	mg/L
2,4,5-Trichlorophenol	95-95-4	400	0.00079	0.00265	400	mg/L
2,4,6-Trichlorophenol	88-06-2	2.0	0.00086	0.00286	2	mg/L
2,4-Dinitrotoluene	121-14-2	0.13	0.00019	0.00062	0.13	mg/L
2-Methylphenol	95-48-7	200	0.00087	0.00288	200	mg/L
3 & 4-Methylphenol	1319-77-3	200	0.00067	0.00223	200	mg/L
Hexachlorobenzene	118-74-1	0.13	0.00022	0.00074	0.13	mg/L
Hexachlorobutadiene	87-68-3	0.5	0.00030	0.00099	0.5	mg/L
Hexachloroethane	67-72-1	3.0	0.00007	0.00024	3	mg/L
Nitrobenzene	98-95-3	2.0	0.00015	0.00051	2	mg/L
Pentachlorophenol	87-86-5	100	0.00089	0.00295	100	mg/L
Pyridine	110-86-1	5.0	0.00176	0.00587	5	mg/L
Surrogates			**Lower**	**Upper**		**Units**
Surr: 2,4,6-Tribromophenol	118-79-6		7	146		% Recovery
Surr: 2-Fluorobiphenyl	321-60-8		32	119		% Recovery
Surr: 2-Fluorophenol	367-12-4		1	123		% Recovery
Surr: Nitrobenzene-d5	4165-60-0		26	132		% Recovery
Surr: Phenol-d5	4165-62-2		1	91		% Recovery
Surr: Terphenyl-d14	98904-43-9		38	126		% Recovery

TCLP METALS
SW 846 METHOD 1311 / 6010B

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
Arsenic	7440-38-2	0.0074	0.0022	0.0074	5	mg/L
Barium	7440-39-3	0.0010	0.0003	0.0010	100	mg/L
Cadmium	7440-43-9	0.0011	0.0003	0.0011	1	mg/L
Chromium	7440-47-3	0.013	0.004	0.013	5	mg/L
Lead	7439-92-1	0.0046	0.0014	0.0046	5	mg/L
Mercury	7439-97-6	0.00015	0.00005	0.00015	0.2	mg/L
Selenium	7782-49-2	0.014	0.004	0.014	1	mg/L
Silver	7440-22-4	0.011	0.003	0.011	5	mg/L

REFERENCE LIMITS AND EVALUATIONS TABLE
Investigative Derived Waste (IDW)

TCLP PESTICIDES
SW 846 METHOD 1311 / 8081A

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
alpha-Chlordane	5103-71-9	0.000030	0.000009	0.000030	NS	mg/L
Chlordane (Technical)	57-74-9	0.000325	0.000102	0.000325	0.03	mg/L
Endrin	72-20-8	0.000020	0.000006	0.000020	0.02	mg/L
gamma-Chlordane	5103-74-2	0.000024	0.000007	0.000024	NS	mg/L
Heptachlor	76-44-8	0.000019	0.000006	0.000019	0.008	mg/L
Heptachlor epoxide	1024-57-3	0.000021	0.000007	0.000021	0.008	mg/L
Lindane	58-89-9	0.000022	0.000007	0.000022	0.4	mg/L
Methoxychlor	72-43-5	0.000020	0.000006	0.000020	10	mg/L
Toxaphene	8001-35-2	0.000562	0.000176	0.000562	0.5	mg/L
Surrogates			**Lower**	**Upper**		**Units**
SURR:2,4,5,6-CL4-m-xylene	877-09-8		25	140		% Recovery
SURR:Decachlorobiphenyl	2051-24-3		30	135		% Recovery

REACTIVITY

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
Cyanide, Reactive	57-12-5	4	NA	NA	150	mg/L
Sulfide, Reactive	18496-25-8	2	NA	NA	500	mg/L

RCRA CHARACTERISTICS

		LABORATORY LIMITS				
Analyte	**CAS #**	**RL**	**MDL**	**PQL**	**DQL**	**Units**
Ignitability		Flashpoint < 60°C			Flashpoint < 60°C	
Corrosivity		pH 0.0 – 14.0			pH <2.0 or >12.5	

LEGEND FOR IDW TABLES

CAS # Chemical abstract number
RL Reporting Limit
MDL Method Detection Limit
PQL Practical Quantitation Limit
DQL Data Quality Limit - based on TCLP standards (SW-846 Chapter 7, Table 7-1) and RCRA characteristics of hazardous waste
NS None Specified
mg/L Milligrams per liter

ATTACHMENT 2
CT Laboratories Quality Assurance Manual

USACE Candidate Environmental Laboratory
Self-Declaration Form

The following self declaration form is provided to the candidate environmental laboratory by the COR, completed by the laboratory and returned to the COR. The self declaration form should also be provided to the laboratory evaluator (if different from COR) with requested documentation that demonstrates compliance with the QSM prior to the provision of environmental analytical support for USACE contracts. Before testing services can be performed by the laboratory, the COR will notify the candidate laboratory of the acceptability of the declaration and supporting documentation. The form is to be updated on an annual basis.

Legal name of laboratory:	**Foree Holdings dba CT Laboratories**
Street address:	**1230 Lange Court, Baraboo, WI 53913**
Name of Owner:	**Molly Foree**
Owner address (if different):	
Phone number:	**608-356-2760**
E-Mail address:	**mforee@ctlaboratories.com**
Web site:	**www.ctlaboratories.com**
Laboratory director:	**David Berwanger**
Phone number:	**608-356-2760**
E-Mail address:	**dberwanger@ctlaboratories.com**
Quality Assurance Officer:	**Dan Elwood**
Phone number:	**608-356-2760**
E-Mail address:	**delwood@ctlaboratories.com**

USACE Candidate Environmental Laboratory
Self-Declaration Form

The undersigned persons understand and acknowledge that:

a. Laboratory operations, which will be utilized for testing in support of environmental analytical testing for USACE, are in full compliance with the most recent version of the DOD Quality Systems Manual (including NELAC Standard Chapter 5 and Appendix requirements). All written documentation provided to USACE, accompanying this declaration, accurately reflect policy and practices implemented by laboratory staff.
b. The Laboratory will notify USACE immediately of change in status of laboratory operations that may affect on-going compliance as declared per item a.
c. The Laboratory acknowledges that USACE may audit the laboratory, relative to policy compliance at any time deemed appropriate; and will allow a designated COR full access to information and facilities to conduct such audit operations.
d. Signatories are authorized to sign this form on behalf of the owner and that there are no misrepresentations in the information provided in the initial laboratory assessment package.

Signature of Quality Assurance Officer:	
Date:	1/7/08
Signature of Laboratory Director:	
Date:	1/7/08

<u>Note:</u> Minimally, the laboratory evaluator should receive the completed declaration form with the following material (to verify compliance with the QSM):

- *A copy of the laboratory's most current Quality Assurance Manual (e.g., the laboratory's ethics program policies and quality systems procedures), and select QA SOPs (minimally, the laboratory's SOPs for MDL studies and LCS control chart limits).*
- *The determinative and preparatory method SOP for the parameters for which testing will be performed.*
- *Method performance demonstration for the parameters of interest – minimally, MDL studies and LCS control ranges for the preparatory determinative method combinations and PT sample results (from a NELAP accredited PT provider) for the parameters of interest (or NELAP accreditation for these parameters, when NELAP accreditation is offered for the parameter of interest).*

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

CT LABORATORIES

1230 LANGE CT.

BARABOO, WI 53913

NELAP ACCREDITED
ACCREDITATION NUMBER #100457

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Ron Turpin
Manager
Environmental Laboratory Accreditation Program

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 002116
Expiration Date: 09/30/2009
Issued On: 09/16/2008

State of Illinois
Environmental Protection Agency

Certificate No.: 002116

Awards the Certificate of Approval

CT Laboratories
1230 Lange Ct.
Baraboo, WI 53913

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Hazardous and Solid Waste, Inorganic

1010

Ignitability

1311

TCLP (Organic and Inorganic)

1312

Synthetic Precipitation Leaching Procedure

6010B

Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Calcium	Chromium
Cobalt	Copper	Iron
Lead	Lithium	Magnesium
Manganese	Molybdenum	Nickel
Potassium	Selenium	Silica
Silver	Sodium	Thallium
Titanium	Vanadium	Zinc

7041

Antimony

7060A

Arsenic

7196A

Chromium VI

7421

Lead

7470A

Mercury

7471A

Mercury

7740

Selenium

7761

Silver

7841

Thallium

9010B

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Environmental Protection Agency

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CT Laboratories
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Hazardous and Solid Waste, Inorganic	*9010B*	Cyanide
9012A		
Cyanide		
9040B		
Hydrogen Ion (pH)		
9045C		
Hydrogen Ion (pH)		
9050A		
Specific Conductance		
9056		
Bromide	Chloride	Fluoride
Nitrate	Nitrite	Phosphate
Sulfate		
9065		
Phenolics		
9095A		
Paint Filter		
Chapter 7/9012A		
Reactive Cyanide		
Chapter 7/9034		
Reactive Sulfide		
Hazardous and Solid Waste, Organic		
8015B		
Diesel range organics (DRO)	Gasoline range organics (GRO)	
8081A		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	alpha-Chlordane
beta-BHC	Chlordane - not otherwise specified	delta-BHC
Dieldrin	Endosulfan I	Endosulfan II
Endosulfan sulfate	Endrin	Endrin aldehyde
Endrin ketone	gamma-BHC (Lindane)	gamma-Chlordane
Heptachlor	Heptachlor epoxide	Methoxychlor
Toxaphene		
8082		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
8260B		
1,1,1,2-Tetrachloroethane	1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene

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Hazardous and Solid Waste, Organic	*8260B*	1,3-Dichloropropane
1,4-Dichlorobenzene	2,2-Dichloropropane	2-Butanone (Methyl ethyl ketone, MEK)
2-Chloroethyl vinyl ether	2-Chlorotoluene	2-Hexanone
4-Chlorotoluene	4-Methyl-2-pentanone (Methyl isobutyl ketone, I	Acetone
Benzene	Bromobenzene	Bromochloromethane
Bromodichloromethane	Bromoform	Bromomethane
Carbon disulfide	Carbon tetrachloride	Chlorobenzene
Chlorodibromomethane (Dibromochloromethan	Chloroethane	Chloroform
Chloromethane	cis-1,2-Dichloroethene	cis-1,3-Dichloropropene
Dibromomethane	Dichlorodifluoromethane	Dichloromethane (Methylene chloride)
Ethylbenzene	Hexachlorobutadiene	Isopropylbenzene
Methyl-t-butyl ether	m-Xylene	Naphthalene
n-Butylbenzene	n-Propylbenzene	o-Xylene
p-Isopropyltoluene	p-Xylene	sec-Butylbenzene
Styrene	tert-Butylbenzene	Tetrachloroethene
Tetrahydrofuran	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	Trichloroethene	Trichlorofluoromethane
Vinyl acetate	Vinyl chloride	Xylenes (total)
8270C		
1,2,4,5-Tetrachlorobenzene	1,2,4-Trichlorobenzene	1,2-Dichlorobenzene
1,2-Diphenylhydrazine	1,3-Dichlorobenzene	1,4-Dichlorobenzene
2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	2,4-Dichlorophenol
2,4-Dimethylphenol	2,4-Dinitrophenol	2,4-Dinitrotoluene (2,4-DNT)
2,6-Dichlorophenol	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol (o-Cresol)
2-Naphthylamine	2-Nitroaniline	2-Nitrophenol
3,3'-Dichlorobenzidine	3-Methylphenol (m-Cresol)	3-Nitroaniline
4,6-Dinitro-2-methylphenol	4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol
4-Chloroaniline	4-Chlorophenyl phenyl ether	4-Methylphenol (p-Cresol)
4-Nitroaniline	4-Nitrophenol	Acenaphthene
Acenaphthylene	Acetophenone	Aniline
Anthracene	Benzidine	Benzo(a)anthracene
Benzo(a)pyrene	Benzo(b)fluoranthene	Benzo(g,h,i)perlyene
Benzo(k)fluoranthene	Benzoic acid	Benzyl alcohol
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether
Bis(2-ethylhexyl)phthalate	Butyl benzyl phthalate	Carbazole
Chrysene	Dibenz(a,h)anthracene	Dibenzofuran
Diethyl phthalate	Dimethyl phthalate	Di-n-butyl phthalate
Di-n-octyl phthalate	Diphenylamine	Fluoranthene
Fluorene	Hexachlorobenzene	Hexachlorobutadiene
Hexachlorocyclopentadiene	Hexachloroethane	Hexachloropropene
Indeno(1,2,3-cd) pyrene	Isophorone	Naphthalene
Nitrobenzene	N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine	N-Nitrosopyrrolidine	Pentachlorophenol
Phenanthrene	Phenol	Pyrene
Pyridine		
8310		

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Hazardous and Solid Waste, Organic

8310

Acenaphthylene	Anthracene	Acenaphthene
Benzo(a)pyrene	Benzo(b)fluoranthene	Benzo(a)anthracene
Benzo(k)fluoranthene	Chrysene	Benzo(g,h,i)perylene
Fluoranthene	Fluorene	Dibenz(a,h)anthracene
Naphthalene	Phenanthrene	Indeno(1,2,3-cd) pyrene
		Pyrene

8330

1,3,5-Trinitrobenzene (1,3,5-TBN)	1,3-Dinitrobenzene (1,3-DNB)	2,4,6-Trinitrotoluene (2,4,6-TNT)
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Amino-4,6-dinitrotoluene (2-Am-DNT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
m-Nitrotoluene (3-Nitrotoluene, 3-NT)	Nitrobenzene	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocin
o-Nitrotoluene (2-Nitrotoluene, 2-NT)	p-Nitrotoluene (4-Nitrotoluene, 4-NT)	

Wastewater, Inorganic

SM5210B,18Ed
Biochemical Oxygen Demand (BOD)

USEPA160.2
Residue (TSS)

USEPA1664RA
Oil and Grease

USEPA350.1R2.0
Ammonia

USEPA351.2R2.0
Total Kjeldahl Nitrogen

USEPA376.1
Sulfide

USEPA410.4R2.0
Chemical Oxygen Demand (COD)

USEPA415.1
Total Organic Carbon (TOC)

CT Laboratories
1230 Lange Ct.
Baraboo, WI 53913
608-356-2760

Quality Assurance Manual

Approved By:

_____ _____

David Berwanger *Date*
Laboratory Director, CT Laboratories

Reviewed By:

_____ _____

Dan Elwood *Date*
Quality Assurance Coordinator, CT Laboratories

Abstract

The purpose of this document is to provide details of the Quality Assurance program for CT Laboratories. The scope includes all practices within the laboratory sections aimed at providing results that are of known and acceptable quality, and ensuring that CT Laboratories and the client's objectives for precision, accuracy, and detectability of analytical results, and reporting are met.

This Quality Assurance Manual (QAM) outlines the general Quality Assurance/Quality Control (QA/QC) measures employed in the following areas:

- Organizational structure and position responsibilities
- Staff training and proficiency demonstration
- Facility and equipment capabilities
- Sample collection and handling
- Sample, process, and document control
- Assessments, method performance, and corrective actions
- Data validation and reporting

Additional details of sample collection, preparation, and analysis procedures, along with method-specific QA/QC requirements, can be found in the separately bound CT Laboratories Standard Operating Procedures (SOPs). In-depth health and safety guidelines are covered in CT Laboratories Health and Safety Plan.

Supplements to the QAM are available to provide more detailed QA/QC guidance specific to a given technique or method. All supplements are included in their entirety with every controlled copy of the QAM document. For purposes of client review, uncontrolled copies of the QAM may include only supplements of interest. Supplements may also be provided to clients as stand-alone references. Topics covered in each supplement are presented in the following format ("x" denotes the letter identifier for a given supplement):

Sx 1.0 Data Quality Objectives
Sx 2.0 Methods Requirements
Sx 3.0 Instrument Testing, Inspection, and Maintenance Requirements
Sx 4.0 Instrument Calibration and Frequency
Sx 5.0 Quality Control
Sx 6.0 Data Management

Laboratory Acronyms, Abbreviations, and Symbols

Acronym	*Description*	*Acronym*	*Description*
%R	percent recovery	**QA**	quality assurance
%RSD	percent relative standard deviation	**QAC**	quality assurance coordinator
CA	corrective action	**QAM**	quality assurance manual
CCV	continuing calibration verification	**QAPjP**	quality assurance project plan
COC	chain of custody	**QC**	quality control
CRDL	contractor required detection limit	**RPD**	relative percent difference
DQO	data quality objective	**RSD**	relative standard deviation
ICAL	initial calibration	**RT**	retention time
ICV	initial calibration verification	**SOP**	standard operating procedure
IDC	initial demonstration of capability	**VTSR**	validated time of sample receipt
IDL	instrument detection limit	**X**	mean value
IPR	initial precision and recovery standard	**°C**	degrees Celsius
LCS	laboratory control sample	**>**	greater than
LOD	limit of detection	**<**	less than
LOQ	limit of quantitation	**%**	Percent
MB	method blank	**+/-**	plus or minus
MDL	method detection limit	**g**	Gram
MS	matrix spike	**kg**	Kilogram
MSA	method of standard additions	**h**	Hour
MSD	matrix spike duplicate	**g/L**	gram per liter
MSDS	material safety data sheet	**L**	Liter
NCR/CAR	nonconformance report/corrective action report	**mg**	Milligram
ng	nanograms	**mg/kg**	milligram per kilogram
NIST	National Institute of Standards and Technology	**mg/L**	milligram per liter
PE	performance evaluation	**mg/mL**	milligram per milliliter
ppb	parts per billion (e.g., µg/L or µg/kg)	**mL**	Milliliter
ppm	parts per million (e.g., mg/L or mg/kg)	**rpm**	revolutions per minute
ppt	parts per trillion	**µg/kg**	microgram per kilogram
PQL	practical quantitation limits	**mg/m^3**	milligram per cubic meter
PRQL	Program-required quantitation limit		

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1.0 Quality Assurance Program

1.1 Quality Assurance Policy

The goal of the CT Laboratories is to use the concept of total quality management to produce analytical results of known, documented, and acceptable quality. The reliability of the data resulting from day-to-day sampling and analysis depends on a strong, effective, and consistently practiced quality assurance (QA) program. The following Quality Assurance Manual (QAM) outlines that program for all aspects of laboratory operations. CT Laboratories objectives for precision, accuracy, and sensitivity of analytical results, and the quality control measures taken in each of these areas are described. These areas include:

- Organizational structure and position responsibilities
- Staff training and proficiency demonstration
- Facility and equipment capabilities
- Sample handling
- Sample, process, and document control
- Assessments, method performance, and corrective actions
- Data validation and reporting

The CT Laboratories QA program is dynamic in nature. It is designed to facilitate the fulfillment of CT Laboratories and the client's goals for its analytical products. It must also satisfy applicable company, local, state, and federal regulatory and program requirements. As such, the QAM is updated as required, based on the results of quality assurance monitoring of analytical processes, internal and external assessments, and changing regulatory policies.

In separately bound supplements to the QAM, more detailed QA/QC guidance is provided, specific to a given technique or method. All supplements are included in their entirety with every controlled copy of the QAM document. For purposes of client review, uncontrolled copies of the QAM may include only supplements of interest. Supplements may also be provided to clients as stand-alone references. Topics covered in each supplement are presented in the following format ("x" denotes the letter identifier for a given supplement):

Sx1.0 Data Quality Objectives
Sx2.0 Methods Requirements
Sx3.0 Instrument Testing, Inspection, and Maintenance Requirements
Sx4.0 Instrument Calibration and Frequency
Sx5.0 Quality Control
Sx6.0 Data Management

CT Laboratories Standard Operating Procedures (SOPs) are also available, which contain specific details of company and method QC practices. SOPs are also updated as required.

The managers and supervisors of CT Laboratories assume responsibility to ensure that all analytical and office staff understand these practices, and their revisions, and implement them immediately. As part of their initial orientation, each laboratory staff member is required to read the QAM and all supplements applicable to their work area. The employee is required to document, through their signature, that they have read, and fully understand the scope of this

Quality Assurance Program
1.1 Quality Assurance Policy
1.2 Data Quality Objectives

document. Signed statements to this effect are placed in the employee's QA training file. Employees are expected to practice policies outlined in these documents as a matter of habit.

CT Laboratories is committed to conducting business ethically and pursuant to the highest standard of honesty and integrity. This requires providing a work environment free from financial, commercial, and other pressures which would adversely affect the quality of work performed by its personnel. Quality control systems described in this manual are in place to help identify analytical quality issues. However, it is the responsibility of each staff member to implement the guidelines of the QAM, and begin the process of corrective action where quality issues are observed. Within the laboratory, both the Laboratory Director and Quality Assurance Coordinator maintain the authority to stop work for issues of quality originating from any of these sources. CT Laboratories is committed to Chapter 5 (Quality Systems) for each test accredited by National Environmental Laboratory Accreditation Program (NELAC), its' accreditation authorities and to the various state and federal agencies that govern environmental testing.

1.2 Data Quality Objectives

The structure of the laboratory's organization and QA program is designed to most effectively and efficiently accomplish established data quality objectives (DQOs) for its analytical services. The quality of measurements made by the laboratory are determined by the following DQOs, or characteristics: representativeness, accuracy, precision, detectability, completeness, and comparability. Specific objectives for each characteristic are generally established to assist in the selection of appropriate sampling and analytical protocols and to identify applicable documentation, sample handling procedures, and measurement system procedures. These quality objectives are established based on site conditions, requirements of the project, and knowledge of available measurement systems, and are addressed whenever appropriate for the data generated.

1.2.1 Representativeness

Representativeness is a qualitative measure of the extent to which a sample acquired from a matrix describes the chemical or physical characteristics of that matrix. Sample collection, handling (e.g., splitting, preservation, storage), and measurements are all conducted according to protocols allowing for the highest degree of representativeness possible for the sample media (air, soil, water, etc.). Recording procedures are utilized which document adherence to proper protocols and maintain sample identification and integrity.

1.2.2 Accuracy

Accuracy describes the degree of agreement between an observed value and an accepted reference (true) value. It includes a combination of random error (precision) and systematic error (bias) components which are introduced in sampling and analytical operations. DQOs for accuracy are established through quality control limits for each parameter measured and for each analytical technique, per matrix where applicable. These objectives are assessed through the analysis of matrix spike/matrix spike duplicates, laboratory control sample, surrogate spike compounds and internal standards, as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits for each parameter and analytical technique are specified in the analytical methods and data validation checklists.

1.2.3 Precision

Precision is a measure of the reproducibility of an analysis under a given set of conditions, regardless of the true value of the target analyte in a sample. The overall precision of a sampling event has both a sampling and an analytical component. DQOs for precision are established through quality control limits for each parameter measured and for each analytical technique, per

matrix where applicable. These objectives are assessed through the analysis of MSDs, LCS duplicates (if available), field duplicates, laboratory replicates, and split laboratory samples, as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits are specified for each parameter and analytical technique in the analytical methods and data validation checklists.

1.2.4 Detectability

Method detectability objectives (commonly referred to as method reporting limits (RLs)) define the lowest concentration or quantities required of the measurement system, for each analyte or parameter. A RL is threshold value below which the laboratory reports a result as "<", with an associated threshold number. The laboratory has established method detection limits (MDLs), limits of detection, (LODs), and limits of quantitation (LOQ) for routine laboratory analytes, any of which may be used as method reporting limits (RLs). Data quality objectives for detectability (i.e., RLs) are established for each parameter measured and for each analytical technique. These objectives are either specified by the analytical method, required by the project, or determined and updated from data acquired through required quality control measurements (e.g., the replicate analyses of samples or standards containing low concentrations of the analyte of concern).

The method RL for an analyte is a function of the specific analytical procedures and can vary substantially as a result of dilutions and similar procedure modifications. In all cases, the method RL necessary to fulfill data quality objectives is confirmed by laboratory measurements. Nominal method RLs for each parameter and analytical technique are listed on the report of analysis.

1.2.5 Completeness

The characteristic of completeness is a measure of the amount of valid data obtained compared to the amount that was expected to be obtained under normal conditions. The amount of valid data expected is based on the measurements required to accomplish project objectives.

1.2.6 Comparability

The characteristic of comparability reflects both internal consistency of measurements and expression of results in units consistent with other organizations reporting similar data. The generation of comparable data requires operating within the calibrated range of an instrument and utilizing analytical methodologies which produce comparable results (e.g., data obtained for phenol by spectrophotometry are not comparable to data obtained for phenol by gas chromatography/mass spectrometry). Appropriate standard units for measurement values are utilized for each measurement system, which yields internally and externally comparable results assuming other comparability criteria are met.

2.0 Organization, Responsibilities, and Training

2.1 Organization

The Baraboo Laboratory facility is comprised of two analytical lab sections: Inorganic and Organic sections. These analytical areas function with support from the Project Management, Sample Receipt, and Data Management groups. Each group is responsible for different processes required to produce the analytical data and report.

2.2 Staff Responsibilities

All laboratory personnel are involved in the QA program. Their level of involvement and responsibilities under the program vary depending on assignment within the unit. In general, laboratory scientists and technicians are responsible for performing and documenting required quality control procedures while they are conducting analyses, whereas laboratory management personnel are primarily involved in monitoring and evaluating results of quality control procedures. Exhibit 2-1 illustrates the organization underlying the following discussion of staff QA responsibilities.

2.2.1 President

The company, including its corporate staff, is headed by the CT Laboratories President. The president functions in a legally binding capacity for all laboratory decisions and operational issues. The President may call upon any of the company management staff or technical staff to perform or assist in special project activities within or outside their normal functional areas, at her discretion.

2.2.2 Laboratory Quality Assurance Coordinator

The Laboratory Quality Assurance Coordinator (QAC) is responsible for the direction of all laboratory QA activities, and reports to the President and Laboratory Director. To promote objectivity in the management and operation of the QA program, the QAC is organizationally independent of the laboratory production functions (Exhibit 2-1). QAC responsibilities include development, documentation, and evaluation of quality assurance/quality control (QA/QC) procedures and policy. The QAC conducts internal audits, reviews data reports, compiles and evaluates method performance, trains staff in QA/QC requirements, tracks nonconformances and corrective actions, prepares quality documents and reports, reviews standard operating procedures, and reports findings and quality issues to the CT Laboratories President and Laboratory Director. A primary responsibility of the QAC is to ensure that all personnel have a clear understanding of the QA program, know their roles relative to one another, and appreciate the importance of their roles to the overall success of the program.

Deputies to act in the Quality Assurance Coordinator's absence include the Laboratory Director and Analytical Group Leader (relative to their particular work areas). The Quality Assurance Coordinator and any deputies performing in a QAC capacity, have the authority to stop any work where quality is deemed questionable.

Organization, Responsibilities, and Training

2.1 Organization

2.2 Staff Responsibilities

2.3 Indoctrination and Training

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Exhibits

2-1 Baraboo Laboratory Organizational Structure

2-2 Technical Training Record

2.2.3 Laboratory Director

The Laboratory Director reports directly to the President. The Director is responsible for the supervision of the following laboratory areas, from which there are direct managerial reports: Business Services, Client Services & Sales, Analytical Group Leaders, Data Management (Information Systems). Exhibit 2-1 shows the functional areas and interfaces within the laboratory.

The Laboratory Director oversees all functional aspects of these systems. Duties may include, but are not limited to, overseeing personnel training, equipment and systems maintenance, laboratory safety, working with customers to identify project-specific requirements, monitoring scheduling and status of work, approval of CT Laboratories Standard Operating Procedures, implementing preventive and corrective actions, and cost control. The Laboratory Director is ultimately responsible for the timely reporting of data and for ensuring that the data meet the client's specifications. Deputies to act in the Laboratory Director's absence include the Analytical Group Leaders and Quality Assurance Coordinator, relative to their particular work areas.

2.2.4 Client Service and Sales Manager

The Client Service and Sales Manager reports directly to the Laboratory Director. Manager of Client Services and Sales directs Project Management, Sample Receiving and Sales. The main objective of this position is developing new client relationships, providing prices to clients, getting costs from subcontracting laboratories and assigning specific clients to Project Managers.

2.2.5 Project Manager

Project Managers report directly to the Client Services and Sales Manager and are assigned specific clients and projects to manage. They are the client's primary point of contact for field and laboratory analytical services. Project managers review analytical results to ensure project data and QC requirements have been satisfied, prepare narrative reports where applicable, and monitor project work so deadlines are met. They are responsible for seeing that clients are informed of any quality problems as soon as possible. Project Managers work directly with the Analytical Group Leaders and laboratory staff involved in their assigned projects to keep staff informed of QA/QC requirements and to monitor work progress. They also work closely with clients to develop work plans and DQOs for current and future work.

2.2.6 Analytical Group Leader

Each Laboratory Section is headed by an Analytical Group Leader (GL), who reports directly to the Laboratory Director. The GL's objective is to ensure the generation of technically valid data through their staff. The GL directly supervises, and is personally involved with, employee training, work load assignments, provides guidance in corrective action situations, oversees internal quality control measures, validates raw data, and manages scheduling within their section.

The Analytical Group Leader is responsible for directing laboratory personnel under their authority to comply with required laboratory quality control practices. This includes monitoring section activities and informing the QA department of any concerns pertaining to data quality. The GL also provides guidance and assistance in the development of new laboratory procedures and associated SOPs. As such, the GL serves as the analysts' first-line resource for technical assistance. The GL is required to evaluate any quality issues observed or reported from the staff in their section, and initiate and document any necessary corrective action, enlisting the assistance of a QAC when needed.

2.2.7 Laboratory Analyst

The Laboratory Analyst reports directly to an Analytical Group Leader for the section in which he or she works. The Laboratory Analyst is responsible for generating technically valid analytical results to be reported on environmental samples, and for documenting all data in support of those results. It is the responsibility of the analyst to follow quality control procedures specified in laboratory standard operating procedures (SOPs), as well as the fulfillment of any special quality control (QC) procedures that are designated for an analysis, and to document any deviations from QC specifications, when conducting sample preparations, analyses, data entry, data reductions and validation.

The Laboratory Analyst also must perform, when applicable, instrument calibration, maintenance and troubleshooting. The Laboratory Analyst may be required to write analytical SOPs at the direction of their group leader, QA Coordinator, or Laboratory Director. They are responsible for critically observing and evaluating all procedures they perform, and for bringing any practices or occurrences that might affect the reliability of analytical data to the attention of the appropriate Analytical GL or QAC. The Laboratory Analyst is required to perform and document any necessary corrective action, enlisting the assistance of their GL or a QAC when needed.

2.2.8 Laboratory Technician

The Laboratory Technician reports directly to an Analytical Group Leader for the section in which they work. The Laboratory Technician performs support work required to produce analytical results. This work may include sample preparation, maintenance activities on equipment (such as ovens, balances, and glassware), data archiving, and other duties, as needed. The Laboratory Technician is required to follow quality control procedures specified in laboratory standard operating procedures (SOPs), as well as the fulfillment of any special quality control (QC) procedures that are designated for an activity, and to document any deviations from QC specifications. He/she is also responsible for critically observing and evaluating all procedures they perform, and for bringing any practices or occurrences that might affect the reliability of analytical data to the attention of the appropriate GL or QAC.

2.2.9 Sample Entry Technician

Sample Entry Technicians reports directly to the Client Service Manager. Their main responsibilities are shipping, receiving and Login.

Sample Entry Technicians primary responsibility is receiving and processing all incoming samples. Received samples and corresponding documentation are carefully reviewed and noted by the Sample Entry Technician. Sample information is entered into the laboratory information management system (LIMS). Logged in samples are labeled with a unique LIMs number and delivered to the lab groups in a timely fashion. On occasion, some samples submitted may require special handling and documentation, where they can be traced through all steps of the laboratory process. In these instances a detailed protocol is followed, where all steps in the laboratory processes are controlled and documented.

The Sample Entry Technician records all discrepancies in sample documentation received with the samples and reconciles the discrepancies with the applicable Project Manager. In addition, specific Login activities include processing samples for subcontracting, processing subcontracting documentation, logging received chemical information into the LIMS, aiding in sample disposal. He/she also maintains bottle inventory, bottle orders, preparation and distribution of collection kits, cooler tracking, and receipt of incoming supplies.

All Sample Entry Technicians are required to follow quality control procedures specified in sample management and laboratory standard operating procedures (SOPs), as well as the fulfillment of any special quality control (QC) procedures that are designated for an activity, and to document any deviations from QC specifications. He/she is also responsible for critically observing and evaluating all procedures they perform, and for bringing any practices or occurrences that might affect the reliability of analytical data to the attention of the Laboratory Director or QAC.

2.3 Indoctrination and Training

The generation of reliable data by a laboratory requires that all operations are conducted by knowledgeable and trained personnel. The laboratory requires the accomplishment of a prescribed sequence of training objectives by a staff member before that individual is designated qualified and permitted to independently conduct any assignment or analyses (SOP FO-11) . The indoctrination and qualification process includes as a minimum:

- Reading and understanding laboratory Quality Assurance Program Plan,
- Reading and understanding applicable laboratory SOPs,
- Reading and understanding applicable reference documents,
- Initially, and annually thereafter, reading and understanding CT Laboratories' Ethics, Integrity and Responsibility Statement.
- Initially, and annually thereafter, participate in Data Integrity training
- Hands-on training by an experienced and qualified individual,
- For analytical methods performing measurements, to successfully demonstrate analytical capability (i.e., four replicate measurements which satisfy precision and accuracy criteria for the method, Section 6.1.3), and
- Continued demonstration of analytical capability by annually performing and obtaining acceptable result on at least one blind sample, for every analytical test that person is certified to perform.

Training records are maintained by the applicable Group Leader, and training files are kept for each staff member in the QA office. A summary of training accomplishments is recorded on a training summary form (Exhibit 2-2).

Exhibit 2-1. CT Laboratories Laboratory Organizational Structure

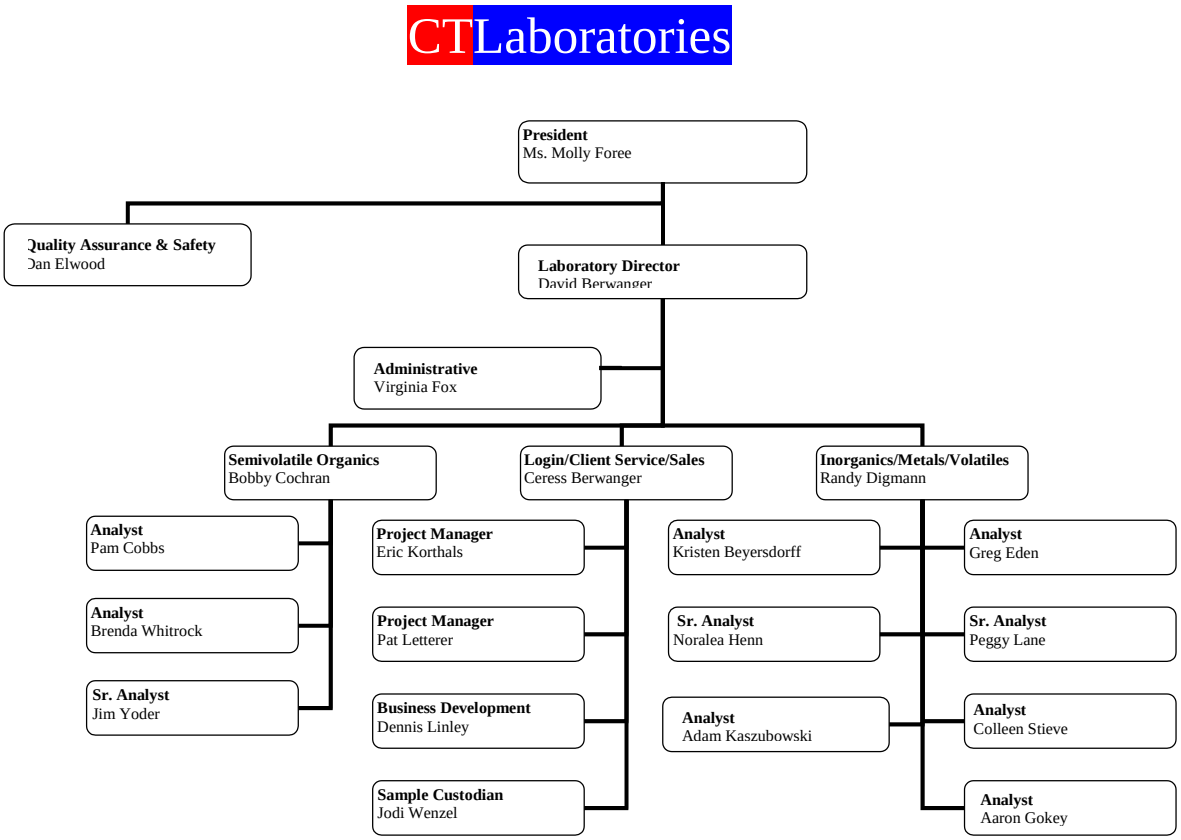

Exhibit 2-2. Technical Training Record

Employee:
Department:
Supervisor:

LABORATORY METHODS

Parameter	Method Reference	Analysis or Prep.	IDCs	Blinds

SUPPLEMENTAL TRAINING

Topic or Area	Date	Documentation

3.0 Facilities, Equipment, and Capabilities

3.1 Laboratory Facilities

CT Laboratories is housed in a 10,800 square foot complex, of which 3,728 square feet is dedicated laboratory space. The laboratory, sample receiving, and administrative office areas are separated, and all are maintained by a full-time physical plant manager and staff.

Six independent heating, ventilation, and air conditioning (HVAC) zones are in place in the laboratories. Zone division is designed to optimize on temperature needs, while minimizing potential cross-contamination. The following table outlines the systems used in each area listed (Table 3.1).

Table 3.1 HVAC System Zoning

Laboratory Area	Number of HVAC Systems
Inorganic Laboratories	1
Organics Laboratories	3
Support Areas	2

Combined laboratory areas (Exhibit 3-1) include 280 linear feet of bench space, 6 fume hoods, 2 walk-in sample storage coolers (secured), 4 incubators, and 8 additional refrigerators. An additional 2,000 square feet of warehouse space is used to support sample storage, sample equipment and secured archives.

The Baraboo facility is operated 365 days a year, employing multiple shifts to enhance turnaround times. Staffing levels are sufficient to support 24-hour shifts for select project workloads.

3.2 Analytical Capabilities and Equipment

3.2.1 Analytical Procedures

The scope of the laboratory's analytical capabilities extends to sampling programs for drinking water, municipal and industrial wastewater, solid/hazardous waste, air, biosolids, and biological tissues. The laboratory performs work on a routine basis for a number of local, state, and federal regulatory programs, including Safe Drinking Water, WPDES/NPDES, RCRA, CWA, UST, etc. The lab also maintains certifications with agencies appropriate to the category of testing. Appendix VI lists the methods and analytes accredited by NELAP. Other federal and state agencies accreditations and certifications are available on request. Table 3.2 provides a detailed listing of testing capabilities and techniques utilized.

Facilities, Equipment, and Capabilities

3.1 Laboratory Facilities

3.2 Analytical Capabilities and Equipment

3.3 Analytical Reagents and Standards

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Tables

3.1 HVAC System Zoning

3.2 Laboratory Testing Capabilities

3.3 Equipment Listing

❖

Exhibits

3-1 Baraboo Laboratory Facility Floor Plan

Table 3.2 Laboratory Testing Capabilities

Traditional Water Quality Parameters by Various EPA Methods		
❖ Spectrophotometry (Colorimetry) ❖ Selective Ion Electrodes ❖ Titrations	❖ Gravimetry ❖ Continuous Flow Analyzer (Autoanalyzer) ❖ Ion Chromatography	
Metals Analysis on Liquids and Solids by EPA Methods 200, 6000, and 7000 Series		
❖ Inductively Coupled Plasma Spectrometry (ICP) ❖ Graphite Furnace Atomic Absorption (AA) Spectrometry ❖ Cold Vapor Generation Atomic Absorption Spectrometry (for Mercury)		
Organics by Gas Chromatography (GC)		
❖ Organics by EPA Methods	8015 Solid Waste 8020 Solid Waste 8081 Solid Waste 8082 Solid Waste	
❖ Hydrocarbon Analysis in Ground Water and Solid Waste State Methods	8015GRO Gasoline 8015DRO Extractables WGRO WI Gasoline Range WDRO WI Diesel Range	
Organics by Gas Chromatography/Mass Spectrometry (GC/MS)		
❖ Organics by EPA Methods	524.2 Drinking Water 8260 Solid Waste 8270 Solid Waste NIST Library Searches	
Organics by High Performance Liquid Chromatography (HPLC)		
❖ Polynuclear Aromatic Hydrocarbons by EPA Methods	610 Wastewater 8310 Solid Waste	
❖ Explosives by EPA / ACOE Methods	8330 Solid Waste 8332 Solid Waste	
Hazardous Waste Characterizations		
❖ Toxicity Characteristics Leaching Procedure (TCLP) ❖ Synthetic Precipitation Leaching Procedure (SPLP)	❖ Paint Filter ❖ Free Liquids ❖ Corrosivity (pH) ❖ Ignitability	

Table 3.2 Laboratory Testing Capabilities *(continued)*

Biosolids Chemical and Biological Testing	
❖ Heavy Metals	❖ Fecal Coliform
❖ Solid, Total and Volatile	❖ Specific Oxygen Uptake Rate (SOUR)
❖ Nitrogen content	❖ Anaerobic Bench Scale Digestions
❖ Aerobic Bench Scale Digestions	❖ UST Bioenumeration
Air Monitoring	
❖ Volatile Organics (VOCs) on Charcoal Tubes	
❖ Heavy Metals on Wipes	
❖ Various Organic Industrial Chemicals	

3.2.2 Equipment Listing

The term "equipment", as used in this manual, includes equipment or instrumentation used in the areas of sample collection, preparation, or analysis. This may include laboratory glassware, as appropriate. The laboratory utilizes all equipment (Table 3.3) as appropriate to standard practice, for a given technique, as specified in a referenced method, or as required by regulatory programs. The equipment investment and subsequent capabilities are sufficient for the analysis of all testing listed in Table 3.2. Instrument redundancy exists for most equipment on-site.

Table 3.3 Equipment List

Instrument	Total No. of Units
GC/MS – Gas Chromatograph/Mass Spectrometers	5
GC – Gas Chromatographs	15
HPLC – High Pressure Liquid Chromatographs	3
ICP	1
AA – Atomic Absorption Spectrophotometer	1
Mercury Cold-Vapor Atomic Absorption Spectrophotometers	1
Colorimetric Autoanalyzers	2
Ion Chromatograph	2

3.2.3 Preventative Maintenance

Manufacturer recommended preventative maintenance schedules must be performed internally for all equipment, in all lab areas. Additionally, some equipment, such as analytical balances, require service checks by the manufacturer. Service calls of this nature are scheduled by the Quality Assurance Coordinator according to the maintenance schedule.

Maintenance logs must be used to document any procedures performed either internally, or by vendor service technicians. These logs must also document maintenance, which may be necessary as a part of corrective action resulting from QC failures. Documentation in the logs is the responsibility of the analyst or technician operating the instrument or equipment.

3.2.4 Equipment Operation

All equipment must be operated according to manufacturer instructions, as detailed in the appropriate CT Laboratories SOPs. The SOPs incorporate the operating instructions, analytical

methodology, and QC requirements for the method performed with the equipment. On occasion, necessary information from the manufacturer's instruction manuals (e.g. tables, lists, etc.) are too massive to reiterate in the CT Laboratories SOP, so direct reference to the manual is allowed.

Internal quality control checks must be performed prior to and during equipment use to monitor and evaluate performance. The frequency of these checks ranges from "per use" to annually, depending on the type of check being performed. Corrective action, which may include maintenance, must be performed if QC checks do not meet SOP or instrument manual specified criteria. QC criteria employed for individual techniques is detailed beginning with Section 8 of this document, and in the applicable SOP.

3.2.5 Equipment Calibration

Equipment requiring calibration must be calibrated according to manufacturer's instructions or the analytical method. General guidelines for analytical instrument calibrations are covered beginning in Section 8 of this manual, and more specifically in the corresponding analytical SOPs.

For equipment where documentation of the calibration can be obtained in the form of hardcopy printouts, the calibration data must be filed with the analytical run data. Where printouts are not possible, the following minimum information must be recorded in a calibration log or on the raw data sheet: equipment identification, calibration date, analyst initials, standard(s) used, certified concentration(s), equipment reading(s) per standard, calibration verification standard(s) results, due date for next calibration (if not "per use").

It is the responsibility of the analyst performing calibration to record this information in the calibration log. Further, when persons who are not CT Laboratories staff perform calibration on any equipment, it is also the responsibility of the analyst to record the details of this work performed, and obtain any applicable certificates from the vendor.

3.3 Analytical Reagents and Standards

3.3.1 Analytical Reagents

Reagents and chemicals used for preservation and analysis must be of ACS-equivalent, or higher, grade. For ultra-trace analysis, higher purity chemicals may be required to reduce interferences, and may be purchased in those instances. Reagents and chemicals must be labeled with the receipt date upon their arrival at the lab. They must be stored according to chemical type and compatibility. Laboratory Group Leaders and analysts using these chemicals are responsible for labeling, proper storage, monitoring of expiration dates, and maintaining a sufficient stock of suitable chemicals (SOP FO-7).

3.3.2 Laboratory-Pure Water

Deionized water must be used to rinse glassware, prepare blanks for analyses, and for routine dilutions of samples, standards, and reagents. The water is produced from a five tank deionizing system, which is composed of a carbon filter tank, a cation bed, an anion bed, and two mixed resin beds. Typically, the resistivity of the product exceeds 2 umho/cm, and it is monitored continuously in the flow path from the resins.

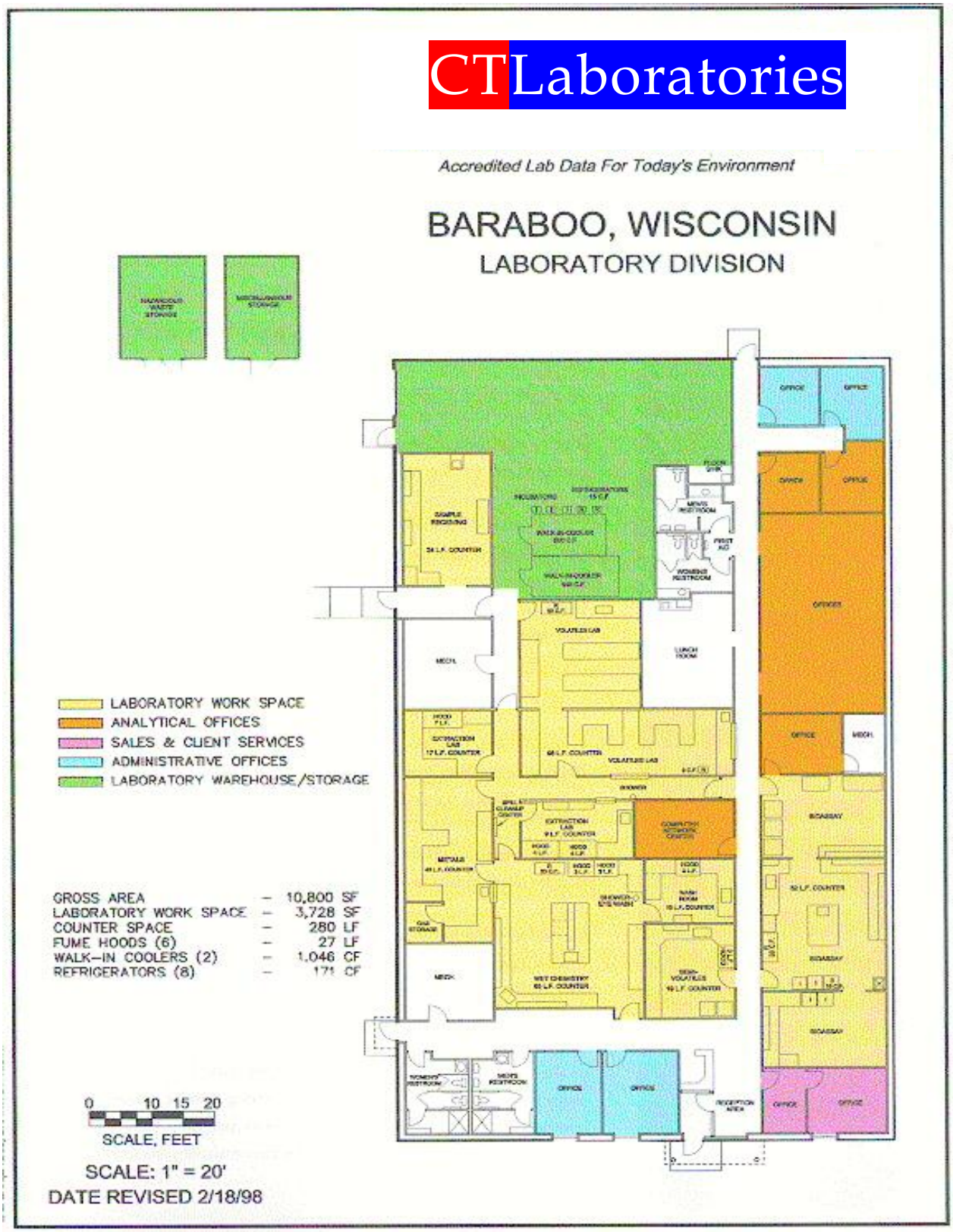
In addition, a secondary scrubbing system for water purification is used at four outlets, and produces water with resisitivity at 18 Mohm. This water is used for all laboratory operations.

3.3.3 Calibration/Reference Standards

While it is possible to prepare many calibration standards in-house, many standards for calibration or calibration verification require NIST traceability, and must be obtained from external sources. All certificates of traceability must be retained on file in the appropriate laboratory section. As an additional measure, analytical checks, spikes, and calibration verification standards must from sources secondary to the calibration standards. Every analytical SOP contains a section on standards, specifying requirements for each analysis.

Standards must be labeled with the receipt date upon their arrival at the lab. The expiration date should be noted, or one assigned and documented on the container, if absent. Standards must be stored separately from other reagents or samples, and the expiration date recorded in a standards logbook with each preparation. Standards exceeding their expiration date must not be used, and must be discarded in accordance with CT Laboratories wastes disposal SOP (FO-8).

Exhibit 3-1. CT Laboratories Laboratory Facility Floor Plan



4.0 Control of Samples

Sample collection, handling, receiving, storage, and associated record keeping procedures are integral parts of the QA program. The policies are designed to ensure that each sample is accounted for at all times. The primary objectives of CT Laboratories sample control procedures are as follows:

- Each sample received for analysis is uniquely identified,
- The correct samples are analyzed and are traceable to the applicable data records,
- Important and necessary sample characteristics are preserved,
- Samples are protected from loss, damage, or tampering,
- Any alteration of samples during collection or shipping (e.g., filtration, preservation, breakage) is documented, and
- A record of sample custody and integrity is established which will satisfy legal scrutiny.

4.1 Sample Collection

Staff at CT Laboratories no longer performs field sampling. Although we do not perform this function it is imperative that we, as a laboratory, understand the process. Field sampling cover the collection and shipping of environmental samples. Samples collected are placed in appropriate containers, having the required preservatives or additives (Appendix I), and labeled with site-specific information to uniquely identify each container at the time of collection. A generic sample label is shown in Exhibits 4-1. Conditions of sampling sites, sample IDs, number of samples, dates/times of collection, equipment calibrations, etc., are recorded on site in field logbooks and / or on chains of custody as appropriate. Samples are stored on ice in chest coolers until their receipt at the laboratory.

Exhibit 4-1. General Sample Container Label

PROJECT NAME		
SAMPLE ID	**SAMPLE DATE**	**SAMPLE TIME**
SAMPLED BY	**PRESERVATIVE**	
ANALYSIS REQUESTED		**___GRAB ___COMPOSITE**

Control of Samples

4.1 Sample Collections

4.2 Sample Custody

4.3 Sample Receiving and Storage

4.4 Sample Distribution and Handling

4.5 Sample Disposal

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Exhibits

4-1 General Sample Container Label

4.2 Sample Custody

All samples remanded to CT Laboratories' custody are documented on a chain of custody (COC), example shown in Appendices II-A. Any information provided by the client (client COCs, special instructions, packing slips, etc.) is recorded on or kept with the COC. The transfer of samples to the laboratory's Sample Management group by CT Laboratories personnel or directly from the client requires a release signature from the representative or client, and an acceptance signature from the laboratory representative receiving the samples, on the COC. The courier service used for shipped samples is noted on the COC at time of receipt. Courier tracking mechanisms serve to document handling of samples prior to receipt at the lab.

4.3 Sample Entry and Storage

The lab Sample Entry Technician verifies that all samples submitted are listed on the COC, and that the COC documentation is complete, prior to signing for the sample delivery group (SDG). Received samples and corresponding documentation are carefully reviewed by the Sample Entry Technician for compliance with regard to condition of containers, sample preservation and temperature, holding times (collection date/time), and accurate identification on the COC (SOPs SS-4 & SS-5).

Once the COC has been verified against the contents of the SDG, sample information is entered into the laboratory information management system (LIMS) by the Login Technician. The LIMS assigns each SDG a unique laboratory number. In addition to the SDG, each sample is given a sample number independent of the SDG. The sample numbers are consecutive, starting at 1, and increase accordingly for all samples. These numbers are used to track all in-house information available on the sample. Login is completed by the end of the day for all samples received on a given day.

After samples are processed they are distributed to various sections of the laboratory for proper storage and analysis. On occasion some samples submitted may require special handling and documentation, where they can be traced through all steps of the laboratory process. In these instances a detailed protocol is followed, where all steps in the laboratory processes are controlled and documented (SOP SS-4).

4.4 Sample Distribution and Handling

The information entered into the LIMS is used to generate status reports showing the analytical work required for a given sample. All samples in-house, logged in through the end of the previous working day are reflected on the status reports. Status reports are printed each morning, and are used by laboratory staff to prioritize sample prep and analysis, and to plan their work schedule for the day.

Restrictive samples (SOP SS-4), retrieved from the sample custodian, must be documented in an internal COC logbook. Personnel removing samples from the storage areas are required to log out the sample numbers removed, date, time, and their initials. Staff must also document in the log the date and time samples are returned to storage. Routine samples not requiring this formal process are removed / returned from designated appropriate storage by the analysts.

Notification of samples arriving with critically short hold times parameters (i.e., less than 48 hours) or special requirements are provided and approved by Project and/or Group Leaders before the receipt or analysis of such samples. It is the responsibility of the analyst to perform the requested analysis within the appropriate hold time or requirements, once notified. Records documenting the notification are retained by the Project Management group.

4.5 Sample Disposal

In general, samples are disposed of 30 days from receipt at the laboratory (21 days for BOD and TSS samples). Arrangements for shorter or longer storage times are made with client approval based on specific project requirements.

Hazardous, or RCRA "D" listed, wastes are returned to the client for disposal. The lab maintains status as a small quantity generator of hazardous waste. Non-hazardous aqueous samples are disposed of by pouring the neutralized sample into a conventional drain to the municipal sewage treatment system. Non-hazardous and special solid wastes are disposed in landfills licensed to accept these wastes, and hauled by licensed special waste haulers. Empty sample containers are recycled by local municipal contracted firms.

5.0 Control of Processes

To ensure the analytical services of the CT Laboratories laboratory satisfy specified quality objectives, the laboratory's processes are carried out under controlled conditions. The controls include the following:

- Laboratory capabilities are defined,
- Work contracts are reviewed prior to accepting work,
- Processes and equipment are approved prior to use,
- Work instructions are documented,
- Criteria for acceptable work and products are defined,
- Work is monitored during a process and verified after completion, and
- Records are kept to document activities.

5.1 Contract Review

The CT Laboratories laboratory process for soliciting and accepting work is designed to prevent the laboratory from accepting work that it does not have either the capability or capacity to perform.

In soliciting analytical service work, the laboratory's capabilities, as described in this document and in the laboratory's Statement of Qualifications, are clearly stated to potential clients. Potential clients are also invited to tour the laboratory and discuss their needs in detail with appropriate laboratory staff. Interested clients are assigned a Project Manager (PM) who works with the client to develop a contract/work plan.

Prior to accepting work, or amending work, a contract is reviewed by the Laboratory Director and applicable Group Leaders to ensure the laboratory has the capability and capacity to perform the work. Determining the schedule for accepting and completing the work is based on the laboratory's current and planned workloads. The Laboratory Director and Laboratory Group Leaders meet weekly to review work status and prioritize, and PMs meet weekly to review status of projects. These meetings keep appropriate personnel informed of laboratory workload and facilitate planning for future work.

5.2 Documents

The CT Laboratories laboratory quality requirements for its analytical services are implemented through this QAM and laboratory standard operating procedures (SOPs).

5.2.1 Key Documents

The CT Laboratories laboratory QAM describes the operational requirements and procedures used by the laboratory to satisfy quality requirements for organization, communication, data, and documentation. Review, approval, and control requirements for the laboratory's QAM are described in Sections 5.2.2 and 5.2.3.

Control of Processes

5.1 Contract Review

5.2 Documents

5.3 Records Management

5.4 Procurement

5.5 Analytical Work Processes

Standard Operating Procedures (SOPs) are used to provide written instruction and implementation detail for operational and analytical functions defined in the laboratory's QAM. These SOPs follow a specific format (outlined in SOP FO-1 *Procedures – Formats and Updates*) that includes, as a minimum, a statement of purpose and scope of the SOP and a step-by-step description of the applicable administrative or analytical process. Review, approval, and control requirements for laboratory SOPs are provided in Sections 5.2.2 and 5.2.3.

5.2.2 Document Review and Approval

All of the laboratory's key quality documents and SOPs receive, minimally, one annual review by the applicable Group Leader, QA Coordinator and / or the Laboratory Director. If it is found that no changes are needed, the reviewer will initial and date the cover page near the document header. Documents and SOPs requiring change follow the procedures outlined in SOP FO-1 (*Procedures – Formats and Updates*) and SOP 1040 (*Document Control*) Note: see next section.

5.2.3 Document Control

All of the laboratory's key quality documents, namely the QAM and SOPs are controlled documents. A controlled document has been through the preparation, review, and approved cycle and may not be changed after release and issue without going through a formal review and change authorization process. Each controlled document contains a document assignment page that assigns the document to a named individual, office, or lab area, indicates the controlled document copy number, and instructs the document assignee on how to maintain the document and enter changes.

Revisions of controlled documents are identified by a consecutive revision number, and the date of the revision on the document title page and page headers within the document. Within one month of final change approval, changes are distributed to those assigned a controlled copy of the applicable document. Each change transmittal is assigned a sequential issue number, which indicates the number of revisions the document has undergone. A record of revisions will accompany each change transmittal to indicate the number and type of changes to the document. Any document designated as an “Uncontrolled Copy” is not subject to updated revisions.

5.3 Records Management

5.3.1 Control of Records

The CT Laboratories laboratory has an established administrative system for the preparation, distribution, filing and archiving of correspondence, records, and data (SOP FO-4). Laboratory records are separated into files based on subject matter. Within the analytical section, subject matter files are essentially the categories of analytical tests performed. Within those categories, the files are further segregated by analysis batch and organized chronologically. The Operations and Project Management sections' files are organized by project/client, then by data reports within a project, and chronologically thereafter.

CT Laboratories laboratory records are maintained in accordance with EPA National Enforcement Investigations Center (NEIC) guidelines. Electronic and magnetic media are physically protected from inadvertent damage or deterioration.

All laboratory paper records are stored in file cabinets within the secure laboratory facility for a period of six months to one year. After than period, the records are placed in labeled boxes and transferred to a locked separate company building. Electronic data are stored in the laboratory's information management system (LIMS) computer (SOP SS-11). Full server backups are performed nightly. Other electronic data include instrument magnetic tapes and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Records are maintained by the laboratory in the event of any change in laboratory ownership. For any unforeseeable event (e.g., no longer in business) all records will be maintained by the laboratory owner.

5.3.2 Disposition of Records

Records are stored for a nominal period of at least five years. Records are stored for longer periods if requested or required by the customer or regulatory authority. Paper records are disposed of through recycling and electronic records are deleted.

5.3.3 Requests for Records

Access to recent (i.e., within the previous year) laboratory records is restricted to laboratory personnel. Access to archived laboratory records is restricted to the Laboratory Director, Analytical Group Leaders, Project Managers, and QACs. All requests for laboratory records should be directed to one of those individuals. Original documents shall not be taken from the file storage area without written permission from one of the listed individuals, and copying and distribution of such documents must also have their authorization.

5.4 Procurement

To maintain efficient, safe, and high quality operations in an analytical chemistry laboratory, it is essential that standardized and clearly understood procedures are used for ordering and receipt of materials and services. Consequently, the CT Laboratories requires its staff to follow specific procurement procedures, which are described in detail in SOP 1100 *Procurement of Materials and Services*. This SOP implements laboratory procurement procedures and describes the practices for source verification, ordering, receiving, inspection and testing, record-keeping, and, if necessary, return to source.

The objectives of the laboratory's procurement procedures are as follows:

- Procurement procedures, including associated documentation, satisfy company and customer requirements
- Timely receipt of requested materials and services
- Received materials and services fulfill intended purposes
- Minimization of costs

5.5 Analytical Work Processes

5.5.1 Control of Analytical Processes

All aspects of laboratory operations are controlled by the key documents, the QAM and SOPs. The SOPs detail and document the procedures which implement the activities and requirements specified in the QAM.

The laboratory uses several means of communication to ensure staff is informed of all quality requirements. Routine operational requirements are communicated to applicable staff through distribution of this QAM and laboratory SOPs. All these documents are controlled internally (see Section 5.2.3) and are issued to selected laboratory staff on an individual basis, depending on staff assignment, task responsibilities, and work location. The QAM and all SOPs are available to all laboratory staff in read only format on the laboratory's computer network. Changes in requirements are communicated to laboratory staff by distribution of revisions to this QAM and applicable SOPs.

Any laboratory staff member observing any occurrence (e.g., equipment failure) that impacts the schedule of deliverables or laboratory capabilities must immediately bring that observation to the attention of their Group Leader and the applicable Project Manager. The PM shall immediately communicate the situation to the affected customer. These communications shall be recorded by the PM in the client's record in laboratory's contact management database, and a copy shall be placed in the project files. Laboratory management staff determines necessary corrective actions for such occurrences at the weekly status meetings, attended by the Laboratory Director, Group Leaders and PMs.

5.5.2 Identification and Traceability of Items

Identification and control of items and materials begins with the procurement process. The laboratory procedures (see Section 5.4) describe the processes for source verification, ordering, receiving, inspection and testing, record-keeping, and if necessary, return to source. Nonconforming items are identified and controlled as described in CT Laboratories SOP 1020, *Nonconformance Identification and Corrective Action*. Items and materials once installed or in use are controlled in accordance with detailed analysis method procedures (SOPs) for the applicable analysis systems (see Section 5.5.1 and all applicable QAM supplements). Samples, standards, and wastes are identified and controlled as described in applicable CT Laboratories SOPs (see Section 4.0).

5.5.3 Computer Hardware and Software

Computer hardware is tested before use and is not used for laboratory work if it does not satisfy manufacturer specifications and laboratory requirements. Likewise, computer software acquired or developed is tested and the test results documented. Computer software installation must be approved and performed by a member of the Information Systems (IS) staff. It is the responsibility of the IS staff member performing the process to document the activity.

6.0 Assessment and Oversight

6.1 Audits and Assessments

Audits and assessments are tools used to examine laboratory systems as they normally operate, and determine if quality assurance needs are being met by current policies.

Audits are used to evaluate and report successes and failures of a system, and offer recommendations for effective improvements or corrections. The laboratory is evaluated through both external and internal auditing procedures. All audits require objective evidence, usually in the form of documentation, to support that stated practices are being followed. Lack of this documentation may be noted as a "deficiency". The audit process involves a review of the system in question, documentation of any deficiencies observed, a debriefing to review the deficiencies, followed by corrective actions and closing report identifying the same.

Assessments are used to evaluate and document specific areas of laboratory and analyst performance. Examples of assessments include an analyst's Initial Demonstration of Capability study, analysis of external and/or internal performance evaluation (PE) samples, calibration curve verifications, etc. Assessment results may be evaluated as part of an audit.

6.1.1 External Audits

External audits are initiated primarily by states, agencies, or affiliations through whom CT Laboratories holds some form of certification (third-party assessments). Audits of this nature cover the entire scope of the accreditation, including sample handling, preparation, analysis, and reporting for all certified parameters. Clients may also perform external audits, or employ a qualified second-party assessor on their behalf. These audits cover the same types of material, but the scope is often limited to parameters of concern to a particular project. The level of detail of an external audit is at the discretion of the auditor.

6.1.2 Internal Audits

Internal audits are conducted by a CT Laboratories Quality Assurance Coordinator (QAC, first-party assessments) (SOP FO-12). An audit may be performed by another designated staff member who is knowledgeable of the process, under the supervision of a QAC, as long as the auditing staff member is not directly performing the process being evaluated. Activities of an internal audit include, but are not limited to:

- Review of the SOP against the referenced method(s)
- Review of the procedure with a staff member who routinely performs the process
- Review of data files for complete and proper documentation, calculations, and quality control frequency (examination may include all testing records showing standardization, spikes, duplicates, and QC samples from one or more analytical runs)
- Review of logbooks for accuracy and completeness
- Review of the process for compliance with company QA policies including error corrections, corrective action, solvent/standard/reagent labeling policies, etc.

Assessment and Oversight

6.1 Audit and Assessments

6.2 Corrective Actions

6.3 Quality Improvement

6.4 Quality Assurance Report to Management

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Exhibits

6.1 Quality Control Charting

CT Laboratories internal audits occur at minimum of one per year. Areas are defined by method or technique for analytical audits, and by section for operational activities audits. Auditing in this manner allows for a comprehensive, on-going review of several areas throughout the year. The scheduling of the audits is at the discretion of the QACs and Laboratory Director.

6.1.3 Demonstration of Capability

An analyst training on a given method must perform an Initial Demonstration of Capability (IDC) study prior to analyzing or reporting client data independently (i.e., without the supervision of a senior analyst)(SOP CL-1). The analyst must prepare four aliquots of a known level of the analyte of interest, analyzed them according to the appropriate method, and demonstrate the ability to recover the analyte within established acceptance criteria. Calculation of IDC results is done through a standard spreadsheet, and may be performed by either the analyst, or a QAC. Results are filed in the employee's technical training file, as well as the IDC file in the QA records.

6.1.4 Performance Evaluations

Performance evaluation (PE) studies involve the analysis of a blind sample (i.e., a sample whose true analyte concentrations and/or analyte identities are not known by the laboratory) for the analyte(s) of interest, followed by evaluation of the results for accuracy by a third party (SOP SS-1). The majority of PEs are performed by the lab in order to maintain state or agency certifications. PE sample analysis may also be required by specific client contract requirements. PE samples may either be provided by the state, agency, or client independently, or ordered by the lab from approved vendors having established PE programs. In-house blind samples may be prepared or purchased and submitted to the lab by a QAC at any time. For routine demonstrations of performance, however, the lab relies on its extensive external PE participation to provide adequate blind performance evaluations.

Upon receipt at the lab, PE sample login is conducted in the same manner as for routine samples. Most PEs are received in the form of concentrates, which must be prepared according to the vendor's instructions in order to obtain an aliquot that is ready for routine sample prep and analysis. The reconstituted aliquot must be prepared and analyzed according to the applicable method along with routine samples. The PE sample results must be subjected to the same QC requirements as used for validating a routine sample result.

All PE raw data and results must be reviewed and approved (initialed and dated) by the appropriate Analytical Group Leader. Copies of raw data and final worksheets, showing the Group Leader's approval with results to be reported, are compiled and filed per method of analysis. Scoring is performed by the provider, and the issued report is retained the QAC files. These reports are available to all staff, auditing agents, and clients upon request.

6.2 Corrective Actions

Any condition that adversely affects compliance with established QC requirements must be identified and corrected as soon as practical. Action taken to correct or preclude the recurrence of that condition is called "corrective action". Some examples of corrective actions include repairs to equipment, revision of an SOP to eliminate a repetitive problem, or obtaining an approved variance to a procedure.

6.2.1 Nonconformances

Nonconformances are items or conditions of a process which do not meet established QAM, SOP, method, or project requirements. As described in CT Laboratories SOP 1020, "Nonconformance Identification and Corrective Action", all nonconformances, and the corrective actions taken as a result, must be documented on a Non-Conformance/Corrective Action Report (CAR). Completion of a CAR should include not only a description of the problem and corrective actions, but also copies of any documentation to support the same. CARs must be routed through the levels of immediate supervisor, Lab Director, and QAC for respective approvals.

Should a nonconformance affect the reportability of a client's data, or the ability to analyze a sample, it is the responsibility of the staff member documenting the nonconformance to notify the Project Manager for that client immediately. The Project Manager must in turn contact the client within two days, describe the details of the problem, act on any further instructions received, and follow up with written notice to the client of the problem and its resolution. A copy of the CAR may be used for this purpose. The CAR must also be signed by the Project Manager in this case.

Client inquiries concerning quality assurance are handled in a similar manner. When a client has a concern regarding laboratory results or procedures, it is the responsibility of the Project Manager to initiate a CAR. The appropriate Group Leader will review testing records for the sample (if applicable) and any circumstances surrounding the complaint. This review may include examination of bench sheets, compiled results (worksheet), the LIMS archives, or applicable log books to check for errors. A copy of the CAR, detailing all findings, will be kept with the file copy of the formal report for the sample in question. Review and approval of the CAR by the Project Manager, Group Leader, the Laboratory Director, and a QAC is required. Again, a written follow-up to the client is required. All CARs are logged and originals retained in QAC files.

6.2.2 Variances

A variance is a type of corrective action involving an approved change to a process or procedure. A variance describes a deviation from a method which affects the operation of the method, but not the method's ability to achieve the performance standards or quality assurance objectives required. Variances must be requested in writing with approvals from the appropriate Group Leader, Laboratory Director, and a QAC (SOP SS-7).

6.2.3 Emergency Alternatives Policy

Under extreme or unavoidable circumstances (such as equipment failure, or irreconcilable matrix difficulties) samples may not be able to be analyzed by methods specified by the client or program (SOP SS-7). Alternative procedures may be acceptable. However, use of these procedures must be approved by the client. Laboratory staff identifying the problem must notify the appropriate Project Manager. The Project Manager is responsible for communicating the situation to the client. This communication must take place prior to reporting the results of the test by the alternate method, and must be documented. The Project Manager may also inform the client if an option exists to sub-contract the samples to an appropriately certified laboratory. Sub-contracting options are also subject to client approval. All information regarding any changes to an existing test for a given sample in the LIMS must be documented.

6.3 Quality Improvement

The CT Laboratories Laboratory Director, QA Coordinator, Client Services and Group Leaders meet weekly to continually assess project work processes, identify needed improvements, assign responsibilities for making improvements, and monitor progress on improvement actions.

The CT Laboratories quality improvement processes are summarized as follows:

- Nonconformance reporting - see Section 6.2
- Corrective Actions - see Section 6.2
- Internal Audits - see Section 6.2.1
- Management assessments - see Section 6.2.1
- Trend Analysis - see Section 6.3.1
- Control Charting - see Section 6.3.2

6.3.1 Trend Analysis

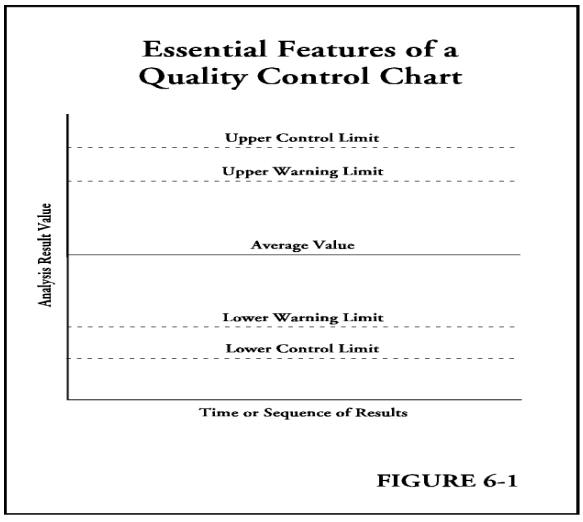
As described in CT Laboratories SOP 1020, "Nonconformance Identification and Corrective Action", the laboratory uses trend analysis to monitor its analytical systems and associated activities. The goals of the trend analysis are as follows:

- To detect quality problems before they become significantly adverse to the quality of the products,
- To allow timely initiation of corrective actions to prevent development of significant quality problems, and
- To ensure continuous quality improvement.

6.3.2 Control Charts

Control charts may be used to monitor trends in analytical performance (SOP SS-3). As illustrated in Exhibit 6-1 a control chart consists of a graph with the vertical axis labeled in units of the analysis or parameter of interest and the horizontal axis labeled in units of time or sequence of results. The upper and lower warning and control limits, which are statistically determined, may be used as criteria for instituting corrective actions. When the parameter being plotted is the relative percent difference (RPD) the lower limits do not apply (i.e., the minimum value of the RPD plotted is always zero and the limits plotted are upper limits).

Exhibit 6-1. Quality Control Charting



A basic principle in a QA program is the establishment of control limits. Such limits are utilized as decision criteria during analytical processes to reduce errors to acceptable levels and statistically characterize the results. Control limits are finite values which are comparable to the measurement values and can be used to statistically assess the acceptability of analytical measurements. There are two goals in establishing control limits. They should yield a narrow enough acceptance range so measurements that lie outside the upper or lower control limit indicate problems within the analytical system (i.e., the system is out of control). The limits, however, should not provide a range so narrow as to cause unnecessary adjustments of the analytical system and rejection of acceptably accurate and reliable results.

CT Laboratories general policy is to utilize control limits where specified by the analytical method or where a sufficient data base exists to establish control limits of ±3s from the mean value of replicate measurements, where "s" is the estimated standard deviation for replicate measurements for the system of concern. Measurements exceeding the control limits (either blank or control sample recovery measurements or precision measurements) usually require halting the analytical process, institution of corrective action measures necessary to obtain acceptable measurements, and documenting the corrective measures taken. This occurrence also normally requires rejection of any results generated between the last acceptable measurement and the unacceptable measurement or reporting those results with the notation that the analytical system was out of control. Warning limits of ±2s may also be utilized. Measurements inside the control limits but exceeding the warning limits require close examination of the measurement system by the analyst. Measurements in this category do not normally require halting the analytical process and rejection of data unless a significant problem is discovered.

6.4 QA Reports to Management

On a weekly basis, a meeting, lead by the Laboratory Director, and attended by the Quality Assurance Coordinator, Analytical Group Leaders, Client Service/Sales Manager and PMs is conducted. One of the purposes of the meeting is to address any unresolved quality issues pertinent to each area of lab operation. Any deficiencies noted through internal or external audits, concerning equipment, systems, training, and/or staffing levels required to maintain or improve product quality, are included. A report detailing the meeting is filed on-line and available to all attendees (SOP FO-12).

In addition, the Laboratory Director, Quality Assurance Coordinator, Analytical Group Leaders and Operations Manager meet monthly to discuss progress and / or closure of all Corrective Action Reports.

7.0 Data Validation, Usability, and Reporting

All processes at CT Laboratories (sample receiving and handling, sample analysis, data reduction, data reporting, data review, etc.) are subject to examination to evaluate adherence to project specifications. This examination consists of several layers of technical and QA review. These reviews ensure that all data released by CT Laboratories were scrutinized by qualified independent reviewers and are scientifically sound, appropriate to the method, and completely documented. Tests performed at CT Laboratories are all performed by EPA or Standard Methods. These methods have been reviewed and found to be EPA Type II methods regarding uncertainty. CT Laboratories statistical quality control data, serves to provide the data necessary data for the uncertainty for these test methods.

All data shall receive analyst review and independent analyst (i.e., qualified peer) review. Analytical Group Leaders, Project Managers, and QA Coordinator also review the data to varying degrees at different points in the review process. These review processes are appropriately documented before data are released from the laboratory.

7.1 Data Review, Verification, and Validation

Data review ensures that raw data are properly collected, reduced, and reported. *Data verification* confirms by examination of the measurement process and provision of evidence, that specified requirements have been met. For example, QC measurements must indicate that deviations between measured values and known values are smaller than the maximum allowable error. *Data validation* is the process of substantiating that specified performance criteria were achieved. At CT Laboratories, a data validation checklist (DVC) for each analytical process outlines the performance criteria for the process. An example DVC is presented in Appendix 3. The checklist is completed and signed for each analysis batch by both the analyst and a qualified peer to document the process.

The CT Laboratories review process must examine as a minimum the following data recording requirements for analyses:

- All original data must be recorded, signed, and dated in blue or black waterproof ink.
- All data must be recorded clearly and accurately in laboratory records, bench sheets, or logbooks, and include applicable sample identification numbers.
- All changes and additions to original data must be made with a single-line strike-out, initialed, and dated by the individual making the change (an explanation of the change or addition must be included if the change or addition deals with rejecting data).
- All data used from logbooks and laboratory records must be transferred and reduced completely and accurately.
- All laboratory records shall be maintained in permanent files.
- Data shall be organized into standard formats.
- All electronic data shall be stored appropriately to ensure that sample and QC data are protected and readily retrievable. Corrections made to hardcopy data must also be made in electronic data files whenever possible.

7.1.1 Data Generator Review and Verification

Data generators (i.e., the analyst or personnel conducting analyses) are responsible for conducting real-time review and verification of 100% of the

Data Validation, Usability, and Reporting

7.1 Data Review Verification and Validation

7.2 Verification Methods and Calculations

7.3 Data Usability

7.4 Data Reporting

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Tables

7.1 Outlier Test

7.2 Analysis Report Deliverables Format

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Exhibits

7-1 Example Data Validation Checklist

7-2 Example Level I Report

data resulting from their activities. This review must be documented by the data generator's signature and review date on the raw data and on a data validation checklist. Data generators are accountable for ensuring that all data they generate are complete, accurate, and compliant with applicable requirements (QAM, SOP, method, or client-specified criteria). Data generators are responsible for performing all data reduction required prior to independent technical review and reporting, and for notifying the appropriate Group Leader, Project Manager, and/or QA Coordinator of any problems encountered during analysis and data review that may potentially impact data quality.

7.1.2 Independent Technical Review and Validation

A minimum of ten percent (10%) of laboratory data must also receive *independent* technical review. The independent technical reviewer(s) must be a qualified individual other than the data generator (e.g., peer analyst or Group Leader). He/she must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The independent reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used.
- Data are reported in the proper units and with the correct number of significant figures.
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations.
- All variances from an accepted method and the rationale for the variations were documented and approved.
- Data were reviewed for transcription errors.
- Analytical data documentation file or data package is complete, including sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary, and completed data validation checklist.
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified.
- Analytical sample holding times were met, or exceptions are documented.

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented on every run sheet, utilizing a data validation checklist. The run sheet is archived in the associated data package.

7.1.3 Project Management Review

One hundred percent (100%) of the data reports must receive a relational technical review by the Project Manager. This review must ensure that:

- Data are technically reasonable based on the technique used.
- Reported analytical data documentation or data package meets the clients' data quality objectives (DQOs) and includes raw data, data forms, calculation records, QC measurement results, narrative comments, COC forms, and sample tags, as appropriate to the report level requested.
- Quality control (QC) criteria (e.g., holding times, spike criteria, etc.) were met, or exceptions are documented.
- Relationships between related parameters are scientifically reasonable.

The PM relational review occurs after the data have been entered into LIMS, and analytical peer review has taken place. This PM review is documented by the Project Manager's signature and date on the final reports, and is done before the reports are released to the client.

7.1.4 Quality Assurance Coordinator Review

Periodically selected data packages are reviewed by the QA Coordinator. This review does not technically validate the data, but rather serves as an overall quality evaluation. This review must ensure that:

- Independent and project management technical reviews were performed as evidenced by the appropriate signature releases.
- Analytical QC documentation is complete.
- Required laboratory QC measurements were properly performed and QC criteria that were not met are documented in a CAR (Section 6.2).
- If the data package is noncompliant with one or more of the project specifications, the QA Coordinator evaluates the nature of the nonconformance. If the nonconformance can be rectified by correcting an error or omission in the data package, the data package is returned to the responsible section for correction. If the nonconformance cannot be rectified by correcting the data package, a CAR must be initiated (Section 6.2).

7.2 Verification Methods and Calculations

During the data review process there are standardized methods and calculations used to examine the measurement process against the specified requirements. These general methods and calculations organized by DQO characteristics outlined in Section 1.2 are described in the remainder of this section and in SOP CL-2.

7.2.1 Representativeness

The appearance and records for samples should, at a minimum, be checked against project requirements for the following:

- Sampling protocols
- Sample types
- Sample containers
- Sample sizes
- Sample numbers
- Sample preservation
- Sample storage
- Sample analysis hold time
- Maintenance of sample chain-of-custody

7.2.2 Accuracy

Accuracy (bias) is a measurement of the extent to which a measured value of a quantity (parameter or analyte) agrees with the accepted value of that quantity. It is assessed by the analysis of samples of known concentration for the analytes of concern.

For LCSs, calibration standards, field reference standards, or additional QC samples of known concentration, accuracy is quantified by calculating the *percent recovery* (%R) of analyte from a known quantity of analyte as follows:

$$\%\,R = \frac{V_m}{V_t} \times 100$$

where:

V_m = measured value (concentration determined by analysis)

V_t = true value (concentration or quantity as calculated or certified by the manufacturer)

A matrix spike (MS) sample or a matrix spike duplicate (MSD) sample is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. A known amount of the analyte of interest is added to a sample prior to sample preparation and instrumental analysis. To assess the effect of sample matrix on accuracy, the %R for the analyte of interest in the spiked sample is calculated as follows:

$$\%\,R = \frac{(SSR - SR)}{SA} \times 100$$

where:

SSR = spiked sample result

SR = sample result

SA = spike added

7.2.3 Precision

Precision is a measurement of the random error in an analytical measurement process. It reflects the degree of agreement between independent measurements determined by the analysis of replicate samples. When calculated for duplicate sample analyses, precision is expressed as the *relative percent difference* (RPD), which is calculated as:

$$RPD\,(\%) = \frac{|S - D|}{(S + D)/2} \times 100$$

where:

S = first sample value (original result)

D = second sample value (duplicate result)

When precision is calculated for three or more replicate determinations, the *relative standard deviation* (RSD), also known as the coefficient of variation, expressed in units of percentage, is used. This is an expression of the spread of the data relative to the mean value of the determinations. The specific formulas used for calculating the RSD are:

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

$$s = \left[\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right]^{1/2}$$

$$RSD\ (\%) = \frac{s}{\bar{x}} \times 100$$

where:

$\bar{x}$ = mean of n measurements

x_i = result value for the i_{th} measurement

n = total number of measurements

s = standard deviation

7.2.4 Method Detection Limits

Method Detection Limits (MDL) or Limits of Detection (LOD) are determined for each analyte for each method used in liquid and solid mediums. These MDLs are determined by (a) conducting replicate analyses of standards at quantities approximately one to five times the estimated MDL, (b) determining the standard deviation, s, of the replicate measurements, and then (c) calculating the MDL from:

$$MDL = t_{(n-1,\ 1 - \alpha = 0.99)} \times s$$

where:

n = number of replicate analyses (For LOD determination the number of replicates is 8)

$t_{(n-1,1 - \alpha = 0.99)}$ = t distribution value appropriate to a 99% confidence level (one-tailed) and standard deviation estimate with n - 1 degrees of freedom

s = standard deviation of the data set

The ratio between the mean spike concentration and the measured MDL should be within one to five for reagent water matrix and one to ten for other matrices. The MDL calculated in this manner, based on 1 % false positive, represents the minimum amount of a substance that can be measured and reported, with 99% confidence that the analyte quantity is greater than zero.

The MDL does *not* represent the analyte quantity for which there is a 99% probability that the analyte will be detected; there is a 50% probability of detection and reporting of the analyte whose actual amount is at the MDL. The analyte quantity at which there is a 99% probability that the analyte will be detected and reported is twice the MDL (1 % false negative).

Because MDLs are usually determined using standards in a clean matrix, they represent optimum obtainable performance. MDLs for actual sample matrices are likely to be higher than those determined using clean matrices.

7.2.5 Quantitation/Reporting Limits

Because of significant uncertainty (±100 %) associated with MDLs determined in a "clean" matrix, plus possible additional variability due to actual sample matrix, CT Laboratories may use higher levels, referred to as "limits of quantitation" or "reporting limits", down to which it routinely reports measured values.

The *limit of quantitation* (LOQ) is defined as 10 times the standard deviation (s) from the MDL determination (see Section 7.2.4). Therefore, the LOQ is roughly 3.33 times the MDL, since the MDL is usually about three times the standard deviation of the data set from the MDL study.

The *reporting limit* (RL), a generic term, is not as rigidly defined as the LOQ. RLs can be either Laboratory RLs, Method RLs, Program RLs, Action RLs or Client Specified RLs. Reporting Limits are usually chosen at a level three to 10 times higher that the MDL. As much as possible, it is also chosen at a level which is below applicable regulatory action levels and which simplifies data review and reporting (e.g., RL of 5.0 µg/L for numerous volatile organic compounds of similar chemical behavior and regulatory action levels).

7.2.6 Completeness

The characteristic of completeness is a measure of the amount of valid analytical data obtained compared to the total number of analyses performed. Valid analytical data are those for which all QC specifications are met. Completeness of the reported data (expressed as a percentage) is calculated as:

$$C\,(\%) = \frac{M_v}{M_t} \times 100$$

where:

M_v = number of measurements judged to be valid (meets all QC specifications)

M_t = total number of measurements performed (based upon number of samples submitted)

7.2.7 Comparability

Comparability of analysis results is evaluated by at a minimum checking the following against project requirements:

- Analysis method utilized
- Analysis QC measurement results
- Units utilized for reporting measurement values

7.2.8 Rejection of Data

Rejection of an analytical result for a sample may be required if established quality control acceptance criteria are not satisfied at any point during the course of analysis. Nominal quality control decision criteria are provided in analytical method SOPs and the corresponding data validation checklists.

Additionally, the CT Laboratories uses a statistical outlier test (*Standard Methods*, 1010 B. Statistics, 17th, 18th, or 19th Editions) for rejection of a questionable value from a group of replicate readings, measurements, results, etc., for an individual sample or standard. Briefly, the test involves dividing the difference between the questionable value and the replicates' mean value by the standard deviation for all replicate values, to calculate a quotient, T. The questionable value is rejected if the calculated T is greater than an established rejection T. The outlier test (Table 7.1) is conducted at the 99% confidence level, which means if the calculated T exceeds the rejection $T_{0.99}$, then the questionable value may be rejected with 99% probability that it is significantly different from the other values.

Table 7.1 Outlier Test for evaluation of a questionable value from a group of replicate values.

Questionable Value[a]	Formula for Calculating T[b]	Number of Values	Rejection Quotient $T_{0.99}$
Smallest value (X_1)	$T = \frac{X_{ave} - X_1}{s}$	3	1.15
		4	1.49
		5	1.75
Largest value (X_n)	$T = \frac{X_n - X_{ave}}{s}$	6	1.94
		7	2.10
		8	2.22
		9	2.32
		10	2.41
		12	2.55
		14	2.66
		16	2.75

a. Arrange values in order of increasing magnitude.
b. If $T > T_{0.99}$ reject questionable value.
X_{ave}= Average value for all replicates.
s = Standard deviation for all replicates, where $s = [\sum(X_n - X_{ave})^2/(n - 1)]^{1/2}$

7.3 Data Usability

Reconciliation of data with DQOs to determine data usability is performed primarily by the applicable Project Managers working in direct communication with the analysts and, if needed, the Group Leader and QA Coordinator. Data which do not satisfy project DQOs may necessitate reanalysis of involved samples or other corrective actions to satisfy the DQOs. If DQOs cannot be satisfied (e.g., no sample available for reanalysis) and data must be reported, an explanation appropriately qualifying the data must accompany the report of analysis. Reference and definitions lists for qualifying flags are provided in Appendix II-A and B.

7.4 Data Reporting

After completion of analyses, sample results are entered into the laboratory's computer-based laboratory information management system (LIMS). After peer review of the data is completed (Section 7.1.2) and the results are acceptable, the results in LIMS are approved and a preliminary report is generated. The applicable Project Manager (PM) reviews the preliminary report (Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections. A final report is then produced. The final report is also reviewed and signed by an appropriate data reviewer before it is submitted to the customer. Each final report has a unique identification number, which is the CT Laboratories Work Order No. listed in the upper right hand corner of the report.

CT Laboratories offers four levels of data reports as illustrated in Table 7.2. For Level II, III, and IV deliverables formats the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management, where the complete package is assembled. The PM reviews the complete package and writes the cumulative analysis, or case, narrative. After final review, approval, and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of 5 years. The retention period may be longer or shorter if requested by the client. These records are stored in the laboratory for approximately twelve months, then transferred to another company building for secure, long term storage.

CT Laboratories provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CT Laboratories LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. The LIMS is maintained on-site by information system personnel. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Table 7.2 Analysis Report Deliverable Formats

Level I:
Analytical Results Surrogate Recoveries – when requested
Level II:
Analytical Results Surrogate Recoveries – where appropriate Method Blank Laboratory Control Sample (LCS) Summary Laboratory Control Sample Duplicate (LCSD) Summary – where appropriate Matrix Spike (MS) Summary Matrix Spike Duplicate (MSD) Summary – where appropriate Replicate/Duplicate – where appropriate Chain-of-Custody documentation
Level III:
Analytical Results Surrogate Recoveries – where appropriate Method Blank Laboratory Control Sample (LCS) Summary Laboratory Control Sample Duplicate (LCSD) Summary – where appropriate Matrix Spike (MS) Summary Matrix Spike Duplicate (MSD) Summary – where appropriate Replicate/Duplicate – where appropriate GC/MS Tune - where appropriate Initial Calibration Continuing Calibration Verification (CCV) Chain-of-Custody documentation
Level IV:
Analytical Results Surrogate Recoveries – where appropriate Method Blank Laboratory Control Sample (LCS) Summary Laboratory Control Sample Duplicate (LCSD) Summary – where appropriate Matrix Spike (MS) Summary Matrix Spike Duplicate (MSD) Summary – where appropriate Replicate/Duplicate – where appropriate GC/MS Tune - where appropriate Initial Calibration Continuing Calibration Verification (CCV) Case Narrative Raw data – including, but not limited to, instrument logs, data sheets, chromatograms, spectra, extraction logs, digestion logs and instrument sequences Chain-of-Custody documentation

Parameter	Matrix	Container	Preservation	Hold Time
Classical Chemistry				
Alkalinity	Aqueous	125 mL plastic	4° C	14 days
Ammonia	Aqueous	250 mL plastic	H_2SO_4 pH< 2	28 days
Ammonia	Solid	4oz cup or 125 mL plastic	4° C	28 days
BOD	Aqueous	1 Liter plastic	4° C	48 hours
CBOD	Aqueous	1 Liter plastic	4° C	48 hours
Chloride	Aqueous	125 mL plastic	4° C	28 days
Chloride	Solid	4oz cup or 125 mL plastic	4° C	28 days
Chlorine, Residual	Aqueous	125 mL plastic	4° C	analyze immediately
Chlorophyll A	Aqueous	1 Liter amber glass	4° C	21 days
COD	Aqueous	125 mL plastic	H_2SO_4 pH< 2	28 days
Conductivity	Aqueous	125 mL plastic	4° C	28 days
Cyanide	Aqueous	500 mL plastic	H_2SO_4 pH< 2	14 days
Cyanide	Solid	4oz cup or 125 mL plastic	4° C	14 days
Fluoride	Aqueous	125 mL plastic	4° C	28 days
Hardness	Aqueous	250 mL plastic	H_2SO_4 pH< 2	6 months
Hexavalent Chromium	Aqueous	125 mL plastic	4° C	24 hours
Hexavalent Chromium	Solid	4oz cup or 125 mL plastic	4° C	28 days
Total Kjeldahl Nitrogen	Aqueous	125 mL plastic	H_2SO_4 pH< 2	28 days
Total Kjeldahl Nitrogen	Solid	4oz cup or 125 mL plastic	4° C	28 days
Nitrate	Aqueous	125 mL plastic	4° C	48 hours
Nitrate	Solid	4oz cup or 125 mL plastic	4° C	48 hours
Nitrate + Nitrite	Aqueous	125 mL plastic	H_2SO_4 pH< 2	14 days
Nitrite	Aqueous	125 mL plastic	4° C	48 hours
Nitrite	Solid	4oz cup or 125 mL plastic	4° C	48 hours
Oil and Grease	Aqueous	1 Liter glass	HCL pH< 2	28 days
Oil and Grease	Solid	4 oz Glass jar	4° C	28 days
Ortho- Phosphate	Aqueous	125 mL plastic	4° C	48 hours
pH	Aqueous	125 mL plastic	4° C	analyze immediately
pH	Solid	4oz cup or 125 mL plastic	4° C	analyze immediately
Phenolics	Aqueous	1 Liter glass	H_2SO_4 pH< 2	28 days
Phenolics	Solid	4 oz Glass jar	4° C	28 days
Available Phosphorus	Solid	4 oz cup	4° C	28 days
Total Phosphorous	Aqueous	125 mL plastic	H_2SO_4 pH< 2	28 days
Total Phosphorous	Solid	4oz cup or 125 mL plastic	4° C	28 days
Total Residue	Aqueous	1 Liter plastic	4° C	7 days
Total Dissolved Solids	Aqueous	1 Liter plastic	4° C	7 days
Total Suspended Solids	Aqueous	1 Liter plastic	4° C	7 days
Total Solids	Aqueous	1 Liter plastic	4° C	7 days
Percent Solids	Solid	4 oz cup	4° C	7 days
Sulfate	Aqueous	125 mL plastic	4° C	28 days
Sulfate	Solid	4oz cup or 125 mL plastic	4° C	28 days
Sulfide	Aqueous	500 mL plastic	NaOH pH > 12/Zinc Acetate	7 days
Sulfide	Solid	4oz cup or 125 mL plastic	4° C	7 days
Turbidity	Aqueous	125 mL plastic	4° C	24 hours
TOC as % Organic Matter	Aqueous	125 mL plastic	4° C	n/a

TOC as % Organic Matter	Solid	4oz cup or 125 mL plastic	4° C	n/a
Metals				
Graphite Furnace Metals	Aqueous	250 mL plastic or 1 L Plastic	HNO_3 pH<2	6 months
Graphite Furnace Metals	Solid	4 oz cup	4° C	6 months
ICP Metals	Aqueous	250 mL plastic or 1 L Plastic	HNO_3 pH<2	6 months
ICP Metals	Solid	4 oz cup	4° C	6 months
Mercury	Aqueous	250 mL plastic	HNO_3 pH<2	28 days
Mercury	Solid	4 oz cup	4° C	28 days
Microbiology				
Total Coliform	Aqueous	100 mL sterile plastic	Sodium Thiosulfate	30 hours
Fecal Coliform	Aqueous	100 mL sterile plastic	Sodium Thiosulfate	30 hours
Fecal Coliform	Solid	100 mL sterile plastic	4° C	30 hours
Fecal MPN	Solid	100 mL sterile plastic	4° C	30 hours
Heterotrophic Bacteria	Aqueous	125 mL plastic	4° C	n/a
Heterotrophic Bacteria	Solid	4oz cup or 250 mL plastic	4° C	n/a
Petroleum Degraders	Aqueous	125 mL plastic	4° C	n/a
Petroleum Degraders	Solid	4oz cup or 250 mL plastic	4° C	n/a
Organics- Volatiles				
BTEX	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4 degree C	14 days
BTEX	Solid	4 oz Glass jar	4° C	14 days
GRO	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4 degree C	14 days
GRO	Solid	4 oz Glass jar	4° C	21 days
Dissolved Gases	Aqueous	40 oz VOA vial	HCl pH<2, 4 degree C	14 days
Methanol Blank	Aqueous	40 oz VOA vial	Methanol	21 days
PVOC	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4 degree C	14 days
PVOC	Solid	4 oz Glass jar	4° C	14 days
Volatiles	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4 degree C	14 days
Volatiles	Solid	Encore	4° C	48 hours to preservation
Organics- Semi-Volatiles				
Semi-Volatiles	Aqueous	1 Liter glass	4° C	7 days to prep
Semi-Volatiles	Solid	4 oz Glass jar	4° C	14 days to prep
DRO	Aqueous	1 Liter glass	4° C	7 days to prep
DRO	Solid	4 oz Glass jar	4° C	14 days to prep
Explosives	Aqueous	1 Liter glass	4° C	7 days to prep
Explosives	Solid	4 oz Glass jar	4° C	14 days to prep
Fingerprint Identification	Aqueous	1 Liter glass	4° C	7 days to prep
Fingerprint Identification	Solid	4 oz Glass jar	4° C	14 days to prep
PAH	Aqueous	1 Liter glass	4° C	7 days to prep
PAH	Solid	4 oz Glass jar	4° C	14 days to prep
PCB	Aqueous	1 Liter glass	4° C	7 days to prep
PCB	Solid	4 oz Glass jar	4° C	14 days to prep
Pesticides	Aqueous	1 Liter glass	4° C	7 days to prep
Pesticides	Solid	4 oz Glass jar	4° C	14 days to prep
TPH	Aqueous	1 Liter glass	4° C	7 days to prep
TPH	Solid	4 oz Glass jar	4° C	14 days to prep

Physical Properties				
Air Filled Porosity	Solid	Shelby Tube		n/a
Corrosivity Index	Aqueous	250 mL plastic	4° C	14 days
Grain Size Analysis	Solid	4 oz cup		6 months
Sieve Analysis P200	Solid	4 oz cup		6 months
Soil Moisture Holding Capacity	Solid	Shelby Tube		n/a
Soil Permeability	Solid	Shelby Tube		n/a
Specific Gravity	Aqueous	125 mL plastic		n/a
Specific Gravity	Solid	4oz cup or 125 mL plastic		n/a
Waste Charaterization				
ASTM Leachate	Solid	4 oz cup	4° C	see test
Cyanide-Reactive	Aqueous	500 mL plastic	NaOH pH > 12	14 days
Cyanide-Reactive	Solid	8 oz glass	4° C	14 days
Flashpoint	Aqueous	8 oz glass	4° C	10 days
Flashpoint	Solid	8 oz glass	4° C	10 days
pH	Aqueous	125 mL plastic	4° C	analyze immediately
pH	Solid	4 oz cup	4° C	analyze immediately
SPLP- Metals	Aqueous	250 mL plastic	HNO3 pH<2	see test
SPLP- Metals	Solid	minimum of 150 grams in glass	4° C	see test
SPLP- Volatiles	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4 degree C	see test
SPLP- Volatiles	Solid	60 mL glass Amber	4° C	see test
SPLP- Semi-Volatiles	Aqueous	(3) 1 Liter Glass	4° C	see test
SPLP- Semi-Volatiles	Solid	minimum of 150 grams in glass	4° C	see test
Sulfide-Reactive	Aqueous	500 mL plastic	NaOH pH > 12/Zinc Acetate	7 days
Sulfide-Reactive	Solid	8 oz glass	4° C	7 days
TCLP- Metals	Aqueous	250 mL plastic	HNO_3 pH<2	see test
TCLP- Metals	Solid	minimum of 150 grams in glass	4° C	see test
TCLP- Volatiles	Aqueous	(3) 40 oz VOA vials	HCl pH<2, 4° C	see test
TCLP- Volatiles	Solid	60 mL glass Amber	4° C	see test
TCLP- Semi-Volatiles	Aqueous	(3) 1 Liter Glass	4° C	see test
TCLP- Semi-Volatiles	Solid	minimum of 150 grams in glass	4° C	see test

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Chain of Custody

Page _____ of _____

Company:
Project Contact:
Telephone:
Project Name:
Project Number:
Project Location:
Sampled By:

Regulatory Program:
UST RCRA SDWA NPDES
Solid Waste Other _____

CTLaboratories

1230 Lange Court, Baraboo, WI 53913
608-356-2760 Fax 608-356-2766
www.ctlaboratories.com

Turnaround Time
Normal RUSH*
Date Needed _____

*Notify Lab prior to sending in RUSH samples. Surcharges:
24 hr 200% 2-3 days 100% 4-9 days 50%, subject to change without notice.

Lab Use Only
Place Header Sticker Here:

Mail Report To:
Company:
Address:
City/State/Zip:

Invoice To:
Company:
Address:
City/State/Zip:

PO No.

Client Special Instructions:

Landfill License Number:

Collection Date	Collection Time	Grab/ Comp	Sample ID Description	Filt? Y/N	WDNR Well ID #	**Matrix:								Total # of Containers	Preservation*	Lab ID #

Fill in Spaces with Bottles per Test

*** Preservation Code**
A=None B=HCL
C=H2SO4 D=HNO3
E=Encore F=Methanol
G=NaOH
O=Other _____

Relinquished By:	Date/Time	Relinquished By:	Date/Time
Received by:	Date/Time	Received for Laboratory by:	Date/Time

Ice Present Yes No
Temperature _____
Cooler # _____

****Matrix**
S–Soil **A**–Air **Sl**–Sludge **M**–Misc Waste
GW–Groundwater **SW**–Surface Water
WW–Wastewater **DW**–Drinking Water

Appendix III. Data Validation Checklist

Data Validation Checklist

Run #:	Method: CYANIDE EPA335.4		Page 1 of 1	
Analysis Date	Analyst / Data Interpretor	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Were samples preserved correctly?	pH >=12 NaOH					
2. Were the samples distilled appropiately?	---					
3. Were the distilled samples accompanied by a prep sheet?	---					
4. Were method blanks prepared at the required frequency?	1per 20 field samples					
5. Was the LCS prepared from a separate source than the calibration standards?	---					
6. Were LCSs prepared at the required frequency?	1per 20 field samples					
7. Were the MS and MSD prepared at the required frequency?	1per 20 field samples					
8. Was the calibration performed using the required number of standards?	Minimum of 3					
9. Was the correlation coefficient acceptable?	>= 0.995					
10. Were the ICV and ICB run immediately after the calibration curve?	---					
11. Were the CV recoveries acceptable?	90 – 110 %					
12. Were the CB results acceptable?	< 0.01 mg/L					
13. Were the CCVs and the CCBs analyzed at the required frequency?	1per 10 analyses					
14. Was the method blank result acceptable?	< 0.1 mg/L					
15. Was the LCS recovery acceptable?	90 – 110 %					
16. Were the MS and MSD recoveries acceptable?	80 – 120 %					
17. Was the RPD between the MS and MSD acceptable?	< 20 %					
18. Were the samples analyzed within holding time?	14 days					

A	Analyte averaged calibration criteria within acceptable limits.
B	Analyte detected in associated Method Blank.
C	Toxicity present in BOD sample.
D	Diluted out.
E	Safe, No Total Coliform detected.
F	Unsafe, Total Coliform detected, no E. Coli detected.
G	Unsafe, Total Coliform detected and E. Coli detected.
H	Holding time exceeded.
J	Estimated value.
L	Significant peaks were detected outside the chromatographic window.
M	Matrix Spike and/or Matrix Spike Duplicate recovery outside acceptance limits.
N	Insufficient BOD oxygen depletion.
O	Complete BOD oxygen depletion.
P	Concentration of analyte differs more than 40% between primary and confirmation analysis.
Q	Laboratory Control Sample outside acceptance limits.
R	See narrative at end of report.
S	Surrogate standard recovery outside acceptance limits due to apparent matrix effects.
T	Sample received with improper preservation or temperature.
U	The analyte was analyzed for but not detected.
V	Raised Quantitation or Reporting Limit due to limited sample amount or dilution for matrix/background interference.
W	Sample amount received was below program minimum.
X	Analyte exceeded calibration range.
Y	Replicate/Duplicate precision outside acceptance limits.
Z	Calibration criteria exceeded.

A Analyte averaged calibration criteria within acceptable limits.

Given the large number of analytes that may be analyzed in some methods, it is likely that some analytes may exceed individual acceptance limit for the Relative Standard Deviation (RSD) for a given calibration. In those instances where the RSD for one or more analytes exceed the individual limit, the calibration is still valid if the average of all analytes is within acceptable limits.

B Analyte detected in associated Method Blank.

For many tests, it is not permissible to have analytes detected in the method blank. However, in some instances it is appropriate to do so. This data flag tells the client that all, or a portion of, the amount found in a sample may be due to laboratory sources. For example, methylene chloride may be found in method blanks for EPA SW846 method 8260 analyses. If methylene chloride is found above the detection limit, in a sample associated with such a blank, the methylene chloride value would then be flagged with a "B" qualifier.

C Toxicity present in the BOD sample.

The presence of toxic compounds, such as some organic compounds, metals, and salts, inhibit the micro-organisms in the sample. In a series of dilutions prepared for the sample, the dilution showing the least toxic effect is chosen to calculate the concentration.

D Diluted out

At times a sample requires dilution in order to overcome a matrix effects or because of a high level of analytes present. This dilution may cause other analytes of interest to be diluted out of range. This qualifier is added to let the data user know normal quantitation is not available.

E Safe, No Total Coliform detected.

Coliform bacteria, a group of indicator organisms, were not found in the sample.

F Unsafe, Total Coliform detected, no E. coli detected.

Coliform bacteria, a group of indicator organisms, were found in the sample. Although E. coli, a type of bacteria harmful to humans, was not found in the sample, conditions exist that indicate the water is potentially dangerous. Boiling water may be advised.

G Unsafe, Total Coliform detected, E. coli detected.

Coliform bacteria, a group of indicator organisms, were found in the sample. In addition, E. coli, a type of bacteria found in excreta of human and other animals that is harmful to humans, was also found. Conditions exist that indicate the water is potentially dangerous and should not be consumed unless boiled.

H Holding time exceeded.

Most EPA and ASTM methodologies have prescribed holding times, within which the sample must be extracted, digested or analyzed. In some cases, samples are received or analyzed by the laboratory past the prescribed holding time. However, such data may still be deemed usable by the data user. In such instances, the client is notified of the occurrence and the sample is analyzed past the holding time, if requested by the client. An "H" qualifier is applied to the flag field to indicate to the data user that the sample was analyzed past the prescribed holding time and the data should be evaluated in that context.

J Estimated value.

Various clients and programs require data reported below a set reporting limit, but above the Method Detection Limit (MDL) to be qualified estimated. Normal statistical confidence may not be applicable within this range. When required, the "J" qualifier is used for this purpose.

L Significant peaks were detected outside the chromatographic window.

Clients may request specific hydrocarbon analyses such as Diesel (DRO) and Gasoline (GRO) analyses. While a sample may or may not contain the hydrocarbon of interest, it may contain significant levels of hydrocarbons that are lighter or heavier than the hydrocarbon of interest. In those cases the result is flagged with an "L" qualifier.

M Matrix Spike and/or Matrix Spike Duplicate recovery outside acceptance limits.

Many EPA methods require that laboratories spike client/field samples to determine potential matrix effects for the analytes of interest. The percent recovery of these spiked analytes can be outside the acceptance limits for various reasons. The most common reasons are that: 1.) the matrix may adversely affect the extraction/digestion and/or analysis of the analyte of interest or 2.) the analytical system is out of control. The laboratory also spikes laboratory water with the same analytes of interest. This is referred to as an LCS sample. A passing LCS indicates the analytical system or process is in control and the anomalous Matrix Spike/Matrix Spike Duplicate (MS/MSD) results are most likely due to matrix effects. In this instance, the MS and/or MSD should be flagged with an "M" data qualifier and the data reported.

If the Matrix Spike recovery is below acceptable limits, it may be likely that the reported results for the associated samples may be underestimated. Conversely, if the Matrix Spike results are high, it may be likely that the reported results for the associated samples may be overestimated. A failing LCS indicates the analytical system is not in control and the sample should be prepared again and reanalyzed. The original data would not be reported in this case

N Insufficient BOD oxygen depletion.

The method calls for a depletion of at least 2 mg/L oxygen for valid readings. In a series of dilutions prepared for the sample, if none of the dilutions deplete by this amount, the results are estimated from the dilution with the greatest oxygen depletion.

O Complete BOD oxygen depletion.

The method calls for a residual concentration of at least 1 mg/L oxygen at the end of five days for valid readings. In a series of dilutions prepared for the sample, if all of the dilutions have less than 1 mg/L residual, then the result is estimated from the dilution with the greatest residual oxygen.

P Concentration of analyte differs more than 40% between primary and confirmation analysis.

When sample results are confirmed the agreement between the quantitative results are evaluated. If one result is significantly higher (>40%) the higher result is reported and the data user alerted by use of the "P" qualifier.

Q Laboratory Control Sample outside acceptance limits.

A Laboratory Control Sample (LCS) is a control matrix (e.g., clean water, clean soil) that has been spiked with the targets of interest, or a subset of those target analytes. The laboratory measures the percent recovery of these spiked compounds to ensure that the analytical system is operating appropriately. By definition, if the LCS recoveries are within acceptance criteria, the laboratory system is in control. If the LCS is out of acceptance criteria, the samples associated with that analytical batch should be prepared again and reanalyzed.

In those instances where it may not be possible to prepare the samples again and reanalyze, the LCS data and associated sample results will be flagged with the "Q" qualifier. If the LCS spike recovery is below acceptable limits, it may be likely that the reported results for the associated samples may be underestimated. Conversely, if the LCS Spike results are high, it may be likely that the reported results for the associated samples may be overestimated.

R See narrative at the end of report

Narratives are added to reports on an as needed bases. This qualifier directs data users to this additional information

S Surrogate standards outside acceptance limits due to apparent matrix effects.

Surrogates measure the preparation and analytical efficiency of an analysis for each sample. This is done by spiking the sample prior to preparation with a compound similar in nature to the target analytes and then measuring the recovery of the surrogate. Surrogates may be outside the acceptance criteria because of matrix affects or system errors. When this occurs and the LCS data are within acceptable limits, the surrogate recovery is flagged with an "S" qualifier.

T Sample received exceeding proper temperature or preservation criteria.

Preservatives are added to samples to protect the integrity of the sample once it has been collected. Adherence to preservation requirements is checked when samples are received at the laboratory. The lack of the proper preservative or the lack of the proper amount of preservative can alter the analytical results for a sample and the data should be evaluated accordingly. Consequently, these results are flagged with a "T" qualifier.

U The analyte was analyzed for but not detected.

Some programs do not allow the usage of the 'less than' sign (<). In these programs this letter is added to the Qualifier column in place of the less than sign.

V Raised Quantitation or Reporting Limit due to limited sample amount or dilution for matrix/background interference.

Laboratory Quantitation or Reporting Limits (RLs) are established in laboratory grade water or in a clean, representative matrix of the sample of interest (e.g., clean soil). However, when samples contain elevated levels of interferences, it becomes impossible to detect target compounds at the normal reporting levels. Consequently, the RL must be raised, whether or not the analytes of interest are detected in the sample. In these cases, sample results, whether below detection or not, are flagged with a "V" qualifier to indicate the RLs have been adjusted due to matrix interferences. Laboratory Quantitation or Reporting Limits (RLs) are established using minimum sample amounts. In the event that the minimum amount is not available, the analyte RL must be elevated accordingly

W Sample amount was below program minimum.

The sample amount provided was less than the amount specified in the analytical method or sampling protocol. The laboratory must rely on the client's sampling staff to provide the laboratory with the appropriate amount of sample. The client is contacted when the sample amount received is less than the amount required.

X Analyte exceeded calibration range.

Given the large number of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the established instrument calibration. In those instances where the calibration curve is exceed, and the analyte can not be reanalyzed, an estimated concentration is provided with an "X" qualifier.

Y Replicate/Duplicate precision outside acceptance limits.

There are many Quality Control (QC) replicates or duplicates that monitor analytical precision. Based on the type of replicate chosen, there are many factors that may affect analytical precision, such as sample homogeneity. In the event that the precision between two analyses is outside the normal acceptance criteria, the data are flagged with a "Y" qualifier to alert the data user to this fact.

Z Calibration criteria exceeded.

The initial standard calibration or calibration verification for the analyte was not within the acceptance criteria of the method.

Absorbance	A measure of the decrease in incident light passing through a sample into the detector.
Accuracy	Accuracy describes the degree of agreement between an observed value (or average of a set of values) and an accepted reference (true) value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations. Accuracy is assessed through the analyses of MS/MSDs, LCS, surrogate spike compounds and internal standards. Results are expressed as percent recovery (%R) and are calculated according to the following formula: $\%R = Result_{meas}/True\ Value \times 100\%$. For MS/MSD sample analyses, the original or "native" sample concentration is taken into account. The following equation is used for the determination of percent recoveries of MS/MSD samples: %R = (Spiked sample result - Sample result)/Amt of Spike Added x 100.
Aliquot	A measured portion or subsample of a field sample taken for analysis.
Analysis	The separation of a compound into its constituent parts, or the breaking down of a complex substance into simpler substances, for the purpose of identification and quantitation of one or more of those components.
Analysis Date and Time	The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.
Analyte	The element, ion, or compound an analysis seeks to determine, which may also be referred to as the target, or target analyte.
Analytical Balance	A mechanical or electronic balance having a sensitivity of 0.1 milligram or less.
Analytical Batch	An Analytical Batch is composed of prepared environmental samples, extracts, digestates or concentrates, which are ***analyzed*** *together as a group.* The length of an Analytical Batch is not limited by number of samples or analyses conducted, but may be limited to a specified time frame, depending on the method. An Analytical Batch can include prepared samples originating from various environmental matrices. An Analytical Batch must contain all associated quality assurance measurements, as required by the CTL QAPP, method, or client contract specifications.
Analytical Method	The sample preparation and instrumentation procedures or steps that must be performed to estimate the quantity of an analyte in a sample.
Analytical Reagent (AR) Grade	Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society.
Analytical Run	A continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the CTL QAPP, method, or client contract specifications.
Analytical Sample	Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibrations, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank. Note that the following are all defined as analytical samples: undiluted and diluted samples, predigestion spikes, duplicates , serial dilutions, analytical spikes, post-digestion spikes, interference check samples (ICS), CRDL standard for AA, CRDL standard for ICP, laboratory control samples (LCS), preparation blanks (PB), method blanks (MB), and linear range analysis sample (LRS).
Arithmetic Mean (Average)	The sum of a set of values divided by the number of values comprising the set.
Audit	An audit is a qualitative evaluation of all components of measurement systems, including the physical facilities for sampling, calibration, and measurement, to determine the adequacy, effectiveness, and compliance with established procedures, instructions, drawings, or other applicable documents. Audits typically involve a comparison of the activities of the laboratory's QA Program Plan with those actually scheduled and performed.

Autozero	Zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.
Background Correction	A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.
Bias	Consistent deviation of measured values from the true value, caused by systematic errors in a procedure.
Blank	A ***Blank*** is an analyte-free matrix carried through a process and designed to monitor the potential introduction of contaminants into that process. For aqueous samples, laboratory-pure reagent water (deionized or distilled) is used as a Blank matrix. Method-specified solvents may also serve as Blanks. Although a universal Blank matrix does not exist for solid samples, it is permissible to use a sample-equivalent aliquot of kiln-fired sand as a suitable Blank. A Blank may be required for any or all appropriate steps of a process. For environmental sampling, this may include: bottle prep, equipment cleaning, sample collection, transport, storage, sample preparation, and/or analysis. The Blank is subjected to the usual analytical measurement process to establish a zero baseline or background value, and is sometimes used to adjust or correct routine analytical results. Specifications of different Blank types are included in this glossary.
Blank, Bottle	A Bottle Blank consists of a blank solution prepared in the lab, placed in a standard sample container containing the same preservative matrix as routinely used when collecting the sample for the specified analyte, and carried through the entire analytical scheme. Bottle blanks evaluate any contamination from containers or preservatives used.
Blank, Calibration	A Calibration Blank consist of a blank solution prepared in the lab, and may be used in the "zeroing", calibration, or calibration monitoring of an instrument. Calibration blanks are used to determine the degree of contamination in the instrumental analysis system, if any. These may also be referred to as instrument blanks.
Blank, Equipment	An Equipment Blank consists of blank solution prepared in the lab, transported to the field, opened, and the contents poured appropriately over or through the sample collection device. The solution is recollected in a sample container, and returned to the laboratory for analysis as a sample. Equipment Blanks permit evaluation of equipment decontamination procedures and potential cross-contamination of environmental samples between sampling locations.
Blank, Field	A Field Blank consists of blank solution prepared in the lab, placed in a sample container in the laboratory or in the field, and treated as a sample in all respects, including preservation, exposure to sampling site conditions, storage, and all analytical procedures. Field Blanks may include equipment blanks, trip blanks, etc. The purpose of the Field Blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.
Blank, Holding	A Holding Blank is blank solution prepared in the lab which is stored and analyzed with samples at the laboratory. Holding Blanks (also known as Storage Blanks) test for contamination in sample storage as well as sample preparation and analysis.
Blank, Laboratory Fortified	A term, equivalent to LCS, used in conjunction with EPA 600/4-88/039m, Method 524.2, which describes an aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The Laboratory Fortified Blank (LFB) is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and measured values in the LFM corrected for background concentrations.
Blank, Trip	Trip Blanks are prepared in the same manner as Field Blanks, and transported with empty sample containers to the site of work, ***except*** that Trip Blanks ***remain sealed*** until analyzed with collected environmental samples. Trip blanks permit evaluation of contamination generated from sample containers, preservatives, or occurring under site conditions or during the shipping and laboratory storage process.

Blind Sample, Single	A performance evaluation sample with a composition known to the submitter, but unknown to the analyst. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
Calibration	Analysis of a series of analytical standards at different specified concentrations, used to define the quantitative response, linearity, and dynamic range of the instrument for target analytes. An analytical curve is established based on the absorbance, emission intensity, or other measured characteristic of known standards. Calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation. The range of the applied calibration should bracket the range of planned or expected sample measurements.
Calibration Curve	The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Standards	A series of known standard solutions prepared from a primary standard solution, including internal standards and surrogate analytes, used to calibrate the instrument response with respect to analyte concentration (preparation of the analytical curve).
Calibration Verification	The periodic analysis of one or more standards (independent of the calibration standards) following calibration, to verify the accuracy of the calibration standards, and that the relationship established in the initial calibration continues to be valid.
Certified Reference Material	A Certified Reference Material (CRM) is a standard in a specified matrix whose analyte values or properties are certified by a technically valid procedure, and which is accompanied by or traceable to a certificate or other documentation is issued by the certifying body.
Chain of Custody	A document designed to trace the custody of a sample(s) from the point of origin to final disposition with the intent of legally proving that custody remained intact and that tampering or substitutions were precluded.
Check Sample	A Check Sample is matrix blank which has been spiked with the target analyte(s) from an independent source to the calibration standards, in order to monitor the execution of the analytical method. The level of the spike shall be at the regulatory action level when applicable. Otherwise, the spike shall be at 5 times the estimate of the quantification limit. The matrix used shall be phase matched with the samples and well characterized: for an example reagent grade water is appropriate for an aqueous sample.
Comparability	Data Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is a significant concern when existing data are being integrated into the data base of an ongoing project, or wherever historical data are being used in support of a project. Comparability is primarily concerned with whether the field sampling techniques, analytical procedures, and concentration units of one data set can be validly compared with another. To ensure comparability, field procedures must be standardized by adhering to SOPs, laboratory procedures must follow the same standard analytical methods, and standard units of measurement must be utilized.
Completeness	Completeness is a measure of whether all information necessary to meet the data quality objectives of a project has been collected. It is defined as the percentage of valid or acceptable results relative to the total number of relevant sample results expected to be obtained under ideal, normal conditions. Valid or acceptable results are those data that meet all acceptance criteria.
Composite Sample	Portions of material collected from more than one spatial location or at different times that are blended and submitted for chemical analysis. Composite samples can provide data representative of a large area with relatively few samples. However, the resulting data are less accurate with regard to the concentrations of contaminants detected in a specific location, because they represent average values.
Concentration	The relative fraction of one substance in another, normally expressed in a weight per volume ratio, weight percent, or volume percent .

Confidence Coefficient	The probability, %, that a measurement result will lie within the confidence interval or between the confidence limits.
Confidence Interval	A Confidence Interval is the set of possible values within which the true value will lie with a specified level of probability. The range includes the upper and lower limits of the interval. It is determined from the standard deviation of a set of analytical values obtained through multiple evaluations for a given analyte. Individual confidence intervals must be calculated for each method or technique, for each matrix. Usually a 95% or 99% confidence coefficient is used for data validation.
Confidence Limit	One of the boundary values defining the confidence interval.
Confirmation	Verification of the presence of a component through a second, separate reanalysis (including prep procedures) of another aliquot of a sample, or by reanalysis using an alternate analytical technique. Alternate techniques may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, additional cleanup procedures, etc.
Continuing Calibration Verification Standard	A Continuing Calibration Verification (CCV) standard is an analytical standard run every 10 analytical samples or every 2 hours, whichever is more frequent, to verify the calibration of the analytical system. The frequency is established based on a reasonable time interval to monitor for drift in the calibration. The concentration level is typically set at mid-range of the calibration curve, but may have other specifications based on method or client contract requirements.
Contract Required Detection Limit (CRDL)	Minimum level of detection for an Analyte that is acceptable under the client contract specifications.
Control Limits	Control Limits define the range within which specified measurement results must fall to be compliant. Corrective action is required if control limits are exceeded, and noncompliant data must be flagged. Courses of corrective actions are dependent on the nature of the exceedence.
Corrective Action	Corrective actions are measures taken to correct conditions adverse to quality and, where necessary, to eliminate the cause in order to prevent recurrence of the problem. Corrective actions involve a systematic problem-solving approach using data to draw conclusions about likely reasons for a problem, and must be documented.
Data Quality Objectives (DQO)	Statements that explain the purpose of collecting the data. DQO's may contain qualitative and quantitative statements that describe the overall level of uncertainty that a decision maker is willing to accept in results derived from environmental data. Dos are determined based on the end uses of the data to be collected.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.
Data Review	An evaluation of laboratory data quality based on a review of method-specific quality control documentation, the CTL QAPP, or as specified in the project-specific laboratory subcontract.
Data Review	The process used to ensure that the proper reduction of raw data has been accomplished.
Data Validation	The act of confirming, through documented evidence, that a process or data set is, in fact, that which is claimed. Data Validation is the process used to confirm that all review and method-specific performance criteria procedures have been completed and met. Validation is documented by signature release.
Defensibility	The degree to which data and its supporting documentation can withstand technical, regulatory, and legal scrutiny.

Deficiency	An unauthorized deviation from acceptable procedures or practices, or a defect in an item. May also be referred to as a non-conformance, finding, or violation.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight of a sample based on it's percent solids, as determined through drying in an oven.
Duplicate	Two representative aliquots (subsamples or splits from the same container) of the same sample matrix subjected to identical analytical procedure independently, in order to assess the precision. The results from duplicate analyses are used to evaluate analytical or measurement precision, but *not* the precision of sampling, preservation or storage internal to the laboratory. Precision is expressed through the calculation of relative percent difference (% RPD).
Duplicate, Field	Field Duplicates are two samples, collected independently, at the same time from the same source and submitted to one laboratory as separate samples. Field duplicate samples are usually composited in the field prior to submittal to the lab, and containerized, handled, and analyzed in an identical manner. The purpose of Field Duplicate samples is to assess the consistency of the *overall* sampling effort and the precision of the entire measurement system, including sampling and analytical procedures. Precision is evaluated based on the relative percent difference (RPD) between the two samples.
Effluent	Effluents may be defined as solid, liquid, or gas wastes which enter the environment as a by-product of man-oriented processes, or the discharge or outflow of water from ground or subsurface storage.
External Standards	External standards are those standards of known concentration which are used to aid in quantitation of data. They may generally include calibration standards, calibration verifications, certified reference materials, etc.
Field Sample	A portion of material received by the lab to be analyzed. A single field sample may be contained in single or multiple containers, depending on container and preservative requirements. All containers of the same sample must be identified by a unique reference.
Frequency	A specification during a prep procedure or analytical sequence allowing for no more than the indicated number of field samples in a prep batch, or no more than the indicated number of analyses between required calibration verification measurements. The indicated number may originate from the CTL QAPP, method, or client contract specifications.
Good Laboratory Practices (GLP)	Either general guidelines or formal regulations for performing basic laboratory operations or activities that are known or believed to influence the quality and integrity of the results.
Grab Sample	An field sample collected from a single location at a specific time. Grab samples must be collected and placed in the appropriate sample containers with no prior mixing.
Headspace	Any area in a container not completely filled by liquid or solid sample, allowing gases to collect in that space.
Holding Time	The storage time allowed between sample collection, extraction, and sample analysis when the designated preservation and storage techniques are employed.
Holding Times	The maximum times that samples may be held prior to analysis and still be considered valid.
Homogeneous	The quality of uniform or representative composition. A homogenous (representative) sample is one which is collected and mixed to ensure representativeness prior to containerizing. Homogenous aliquots are also required to be used by the lab in extraction and analysis. Homogenization is not suitable for volatile organic samples, as mixing will deplete the levels of volatiles present.
Independent Standard	A standard that is composed of analytes from a different source (I.e., different manufacturer) than that used for initial calibration standards.

In-house	Within the laboratory's facility.
Initial Calibration Verification Standard	An Initial Calibration Verification (ICV) standard is an analytical standard prepared from a source independent of that used for calibration, run at the beginning of every analytical run, to verify the calibration of the analytical system. The concentration level is typically set at mid-range of the calibration curve, but may have other specifications based on method or client contract requirements.
Initial Demonstration of Analytical Capability	Initial Demonstration of Analytical Capability (IDC) is a procedure to establish the ability of an analyst, method, or technique to generate acceptable accuracy and precision. The procedure includes the addition of a specified concentration of the target analyte to each of four separate aliquots of laboratory pure water (IPRs). These are carried through the entire analytical procedure, and the percentage recovery and the standard deviation of the results are determined and compared to specified limits.
Initial Precision and Recovery Standards	Initial Precision and Recovery (IPR) standards are four aliquots of a standard at a prescribed concentration, are analyzed to initially establish the ability of an analyst, method, or technique to generate acceptable precision and accuracy. An IPR demonstration is performed the first time a method is used, any time the method or instrumentation is modified, and to certify a new or cross-training analyst on the procedure.
Instrumental Detection Level (IDL)	The analyte concentration that produces a signal greater than five times the signal/noise ratio of the instrument. This is determined by analysis of a standard solution of the analyte in reagent water (not processed through any prep methods) at a concentration of 3x-5x the estimated. IDL, measured on three nonconsecutive days, with seven consecutive measurements per day. The standard deviation obtained for these measurements is then multiplied by three to obtain the IDL concentration.
Interferents	Substances that affect the analysis for the compound or element of interest with either a positive or negative bias.
Internal Standards (IS)	Use of Internal Standards (IS) provides a method for quantifying chromatographic data, and permits correction for inefficiencies. A known amount (concentration) of one or more IS standard compounds is added to a sample prior to preparation and analysis. They are added to every standard blank, sample, matrix duplicate, matrix spike duplicate, etc. Quantitation of the target compounds is made by comparing the peak areas (or heights) of the targets to the areas of appropriate closely-eluting "added" internal standards. Response factors for each sample target relative to an appropriate internal standard are required, and are obtained by analysis of standard solutions containing the organic components and the internal standards.
Laboratory Control Sample	A Laboratory Control Sample is an uncontaminated, interference-free aliquot of sample matrix spiked with known amounts of analyte(s) from a source independent of the calibration standards. The LCS is analyzed using the same sample preparation, reagents, and analytical method as for samples. An LCS must accompany each batch of 20 or fewer prepared samples, per matrix. Aqueous LCSs are prepared using laboratory pure water, while soil/solid LCSs may be prepared using either standardized kiln fired sand, ACS reagent grade sodium sulfate, or a certified reference material (CRM). It's primary function is to verify that analysis is being performed in control. An LCS may also be used to establish intra-laboratory or analyst specific precision and bias.
Laboratory Pure Water	Distilled or deionized water or Type II reagent water which is free of contaminants that may interfere with the analytical test in question.

Level of Detection (LOD)	The analyte concentration in reagent water that produces a signal 2(1.645)s above the mean of blank analyses. This sets both Type I and Type II errors at 5%. May also be referred to as "lower level of detection" (LLD).
Level of Quantitation (LOQ)	The analyte concentration that produces a signal sufficiently greater than the blank that it can be detected within specified levels by laboratories during routine operating conditions. Typically it is the concentration that produces a signal 10s above the reagent water blank signal.
Linear (Dynamic) Range	The concentration range over which the analytical curve (instrument response) remains linear.
Log-in	The receipt and initial management of a sample or group of samples. Log-in generally involves acknowledging receipt at the lab by signing the chain of custody, documenting preservation characteristics, analyses requested (including methodology and/or special instructions), and entering the appropriate sample, reporting and invoice information into the laboratory information management system (LIMS), where it is assigned a discreet in-lab identification number or bar code.
Matrix	The predominant material of which the sample to be analyzed is composed. A sample matrix maybe either waste water, drinking water, soil/sediment, sludge, paint, oil, solid, etc. NOTE: "matrix" is not synonymous with "phase" (liquid or solid).
Matrix Interference	The influence of the sample matrix or sample components upon the ability to qualitatively identify and/or quantitatively measure analytes in environmental samples.
Matrix Spike	A Matrix Spike (MS) is prepared by adding a known amount of target analyte to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the sample's matrix on a method's recovery efficiency for that analyte. Spikes may also be referred to as Laboratory Fortified Matrix (LFM) samples.
Matrix Spike Duplicate	A Matrix Spike Duplicate (MSD) is a second aliquot of the same sample as the matrix spike (MS), prepared and analyzed in the same manner as the MS, in order to determine the precision of the method. An MSD is a replicate of the MS. The relative percent difference (%RPD) between the MS and MSD results is calculated to estimate this precision.
Matrix, Air	Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix.
Matrix, Aqueous	Any water sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Matrix, Biological Tissue	Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
Matrix, Chemical Waste	A product or by-product of a industrial process that results in a matrix not previously defined.
Matrix, Drinking water	Any aqueous sample that has been designated a potable or potential potable water source.
Matrix, Non-Aqueous Liquid	Any organic liquid with <15% settleable solids.
Matrix, Solids	Includes soils, sediments, sludges and other matrices with >15% settleable solids (>10% Total Solids for landfarming applications).
Method Detection Level (MDL)	The method detection limit is defined as the minimum concentration of a substance that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a givin matrix containing the analyte. (Ref.-40CFR136 App. B).

Method of Standard Additions (MSA)	The Method of Standard Additions (MSA) may be required to compensate for matrix effects, but will not counteract spectral effects.. This technique should not be used for interferences which cause baseline shift. The MSA involves the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size from the same sample. The technique involves the analysis of the "unknown" sample and "unknown-plus-known-amounts of standard" with extrapolation of this internal calibration curve to the baseline. Measurements are made on the original sample and after each spike addition. The slope, x-intercept and y-intercept are determined by least-squares analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Also referred to as Standard Additions.
Objective Evidence	Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified.
Percent Solids	The determination of the total dry solids in a sample aliquot (typically for soil samples) by drying a pre-weighed original aliquot in an oven at 105^0, measuring the weight after cooling, and expressing the dried weight as a percent of the wet weight. The percent total solids (%TS) is used to calculate expressions of individual analytes in the sample on a dry weight basis.
Performance Evaluation (PE) Sample	Generally, a single-blind sample submitted to the laboratory through a certifying agency or per client contract specifications. Inter-laboratory comparisons may be used in evaluating the performance, rather than recoveries against analyte true values in the PE sample.
Post-Digestion Spike	The Post-Digestion Spike (PDS) resembles a single-point Method of Standard Additions technique, with the addition of a known amount of standard to a single additional sample aliquot after digestion.
Practical Quantitation Limit (PQL)	The practical quantitation limit is the lowest level that can be *reliably* achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The PQL is highly dependent upon the sample matrix, and is by definition higher than the MDL.
Precision	Precision is a measure of the reproducibility of an analysis under a given set of conditions, regardless of the true value of the target analyte in a sample. The overall precision of a sampling event has both a sampling and an analytical component. Precision data will be assessed from the analysis of MSDs, LCS duplicates (if available), field duplicates, laboratory replicates, and split laboratory samples. Precision is expressed as either standard deviation or relative percent difference (RPD). RPD is calculated according to the following equation, where A and B represent duplicate sample results: RPD (%) = [A - B]/ ([A+B]/2) x 100
Prep Log	An official record of the sample preparation (digestion).
Preparation Batch	The Preparation (Prep) Batch is defined as a group of </= 20 field samples of similar matrix that are prepared together by the same person(s) using the same equipment, with the same method sequence, and same reagent lots, within the same period. The Prep Batch will contain all of the appropriate number and type of QC samples, as specified by the CTL QAPP, method, or client contract specifications.
Preservation	Methods used to retard degradation of chemical analytes within samples by inhibiting decomposition from biological action, chemical reactions, and reducing sorption effects. Methods include limiting headspace, chemical, acid, or base addition, protection from light, cooling, etc.
Procedure	A document that specifies or describes how an activity is to be performed. The document contains detailed, step-by-step descriptions of the sequence of actions to be followed in order to perform a given task. If followed in sequence, a procedure provides enough information that a trained person could complete the covered task without additional information. May also be referred to as a "protocol".
Qualitative Analysis	An analysis to determine the presence, absence or identity of a target analyte.
Quality Assessment	Procedure for determining the quality of laboratory measurements by use of data from internal and external quality control measures, including audits and PE results.

Quality Assurance (QA)	A system of documented procedures, measurements, audits, and corrective actions to ensure that all technical and reporting activities produce legally defensible data with a known and stated level of confidence.
Quality Assurance Manual (QAM)	A CTL document stating the quality policy, quality system and quality practices of the company. This may be also called a Quality Assurance Plan, Quality Plan, or Quality Manual. NOTE: The QAM may include by reference other documentation relating to the laboratory's quality arrangements.
Quality Control (QC)	Those quality assurance activities that specifically measure the performance of a process against defined standards in order to verify that it meets stated requirements. The set of measurements within a sample analysis methodology including calibration, CV/CB, MB, LCS, spikes, duplicates, etc. are examples of quality control activities (QCs). They are used to assure that the prep and analytical process is in control. QC standards are defined by the CTL QAPP, and method or client contract specifications.
Quality System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization, ensuring quality in its work processes, products, items, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities.
Quantitative Analysis	An analysis to measure or determine the amount of a target analyte with measurable precision and accuracy.
Random Error	The deviation in any step in an analytical procedure that can be treated by standard statistical techniques.
Raw Data	Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof, that are necessary for the reconstruction and evaluation of the report, activity, or study. Raw data may include photography microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.
Recovery	The numerical ratio of an amount of analyte measured by the laboratory method to that of the known amount (true value) of analyte present in a sample. Usually expressed as a percent recovery (%R).
Relative Percent Difference	A measure of precision that is calculated as the difference between two results, relative to their arithmetic mean, expressed as a percent.
Relative Standard Deviation (RSD)	Relative Standard Deviation (RSD) is a measure of precision that is calculated as the standard deviation(s) of a *set* of values, relative to their arithmetic mean (x), expressed as a percent. May also be referred to as "coefficient of variation".
Repeatability	The precision of repeated measurements made on subsamples of the same sample at the same laboratory at different times. This measurement may also be referred to as "reproducibility".

Replicate	Any number of representative aliquots (subsamples or splits from the same container) of the same sample matrix subjected to identical analytical procedure independently, in order to assess the precision. Duplicate analyses represent the smallest number of replicates (two) that can be performed. Precision is expressed through the calculation of average relative percent difference (% RPD).
Representativeness	Representativeness is the degree to which a field sample reflects a characteristic of the population of samples taken at a specific location and under a given set of environmental conditions. It may also be seen as a qualitative measure of the extent to which a sample(s) acquired from a medium describe the chemical characteristics of that medium.
Rounding Rules	The following are CTL-employed rules for the rounding of data: *A*. If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44. *B*. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45. *C*. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42. *D*. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations, only then the final answer is rounded to the proper number of significant figures.
Sample Delivery Group (SDG)	A set of field samples received on the same day and at the same time that are grouped together on a chain of custody (COC) for purposes of analysis, tracking, and reporting. SDGs may have more specific definitions (number of samples, by matrix, etc.) depending upon the client contract specifications.
Sample Number	A unique identification number that is designated at the lab for each sample. The sample number appears in all references to the sample, on pertinent sample containers, raw data, reports, and invoices. The chain of custody must reference any field-assigned identifiers relative to the sample.
Sediment	Solid material settled from suspension in a liquid.
Sensitivity	The capability of a method or instrument to discriminate between small differences in analyte concentration. Sensitivity is usually expressed as the slope of the analytical curve, where the slope defines the functional relationship between instrument response and concentration.
Serial Dilution	When a new or unusual matrix is encountered, a series of tests is recommended prior to release of results to verify that no matrix effects are occurring. A 1:4 dilution (1:5 for CLP) is recommended to be run on the sample where the background concentration of the analyte is 10X IDL, with results from the diluted analysis agreeing within + 10% of the original determination.
Sludge	Solid, semisolid, or liquid waste generated from a municipal, commercial, or industrial waste treatment facility or wastewater treatment plant, waste supply treatment plant, or air pollution control facility exclusive of treated effluent from a wastewater treatment plant.
Soil	A natural aggregate of mineral grains, with or without organic materials, that can be separated by mechanical means.
Solution	A liquid mixture of two or more substances where one is dissolved in the other.
Solvent	Liquid that is capable of dissolving another substance. Solvents are used in analytical procedures to extract target analytes from sample matrices. Solvents may also be present in field samples due to their use in a number of manufacturing/industrial processes including the manufacture of paints and coatings for industrial and household purposes, equipment clean-up, and surface degreasing in metal fabricating industries.
Standard Deviation	The square root of the variance of a set of values.

Standard Operating Procedure (SOP)	A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed, and which is accepted as the method for performing certain routine or repetitive tasks.
Statistically Significant	When the difference between a predicted and an observed value is so large that it is improbable that it could be attributed to chance.
Stock Standard	A standard solution which can be diluted to derive other standards of lower concentration, for the purposes of calibration, calibration verification, or method performance evaluation.
Surrogate	A Surrogate is a pure organic compound similar in chemical composition to analytes of interest, but which are not normally found in environmental samples (brominated, fluorinated, or isotopically labeled compounds). Surrogate standards are typically only run with organic (GC, HPLC, and GC/MS) analyses. They are added to EVERY blank, sample, MS, MSD, DUP, standard, etc. in order to evaluate the extraction and analytical process efficiency by measuring recovery. They may also provide additional reference chromatographic information for each sample matrix encountered.
Tentatively Identified Compounds (TICs)	Non-target compounds identified by GC/MS mass spectral library searches. These reported concentrations have a higher associated uncertainty than the reported target analyte concentrations.
Traceability	The ability to trace the history, development, application, or location of an entity by means of an unbroken chain of recorded identifications. In a calibration sense, Traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for quality of the project.
Trend Analysis	A process whereby performance data are collected, organized, displayed, and evaluated for changes over a period of time. Trend analysis is often an integral part of evaluating control chart information.
Validated Time of Sample Receipt (VTSR)	The date and time at which a sample or group of samples is received at the laboratory, as recorded on the COC, the shipper's delivery receipt, or the sample traffic report documentation. The VTS is used to determine sample holding times and data report due dates.
Variance	A measure of the dispersion of a series of results around their average. It is the sum of the squares of the individual deviations from the average of the results, divided by the number of results minus one.
Wet Weight	The weight of a sample aliquot including any original moisture (undried).

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

CT LABORATORIES
1230 LANGE CT.
BARABOO, WI 53913

NELAP ACCREDITED
ACCREDITATION NUMBER #100457

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Ron Turpin
Manager
Environmental Laboratory Accreditation Program

Scott D. Siders
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 001867
Expiration Date: 09/30/2008
Issued On: 10/04/2007

Page 1 of 5

State of Illinois
Environmental Protection Agency

Certificate No.: 001867

Awards the Certificate of Approval

CT Laboratories
1230 Lange Ct.
Baraboo, WI 53913

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

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Hazardous and Solid Waste, Inorganic

1010
Ignitability

1311
TCLP (Organic and Inorganic)

1312
Synthetic Precipitation Leaching Procedure

6010B

Aluminum	Antimony	Arsenic
Barium	Beryllium	Cadmium
Calcium	Chromium	Cobalt
Copper	Iron	Lead
Magnesium	Manganese	Molybdenum
Nickel	Potassium	Selenium
Silver	Sodium	Thallium
Vanadium	Zinc	

7041
Antimony

7060A
Arsenic

7196A
Chromium VI

7421
Lead

7470A
Mercury

7471A
Mercury

7740
Selenium

7761
Silver

7841
Thallium

9010B
Cyanide

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Hazardous and Solid Waste, Inorganic	*9012A*	
Cyanide		
9040B		
Hydrogen Ion (pH)		
9045C		
Hydrogen Ion (pH)		
9056		
Bromide	Chloride	Fluoride
Nitrate	Nitrite	Phosphate
Sulfate		
9065		
Phenolics		
9095A		
Paint Filter		
Chapter 7/9012A		
Reactive Cyanide		
Chapter 7/9034		
Reactive Sulfide		
Hazardous and Solid Waste, Organic		
8015B		
Diesel range organics (DRO)	Gasoline range organics (GRO)	
8081A		
4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	alpha-Chlordane
beta-BHC	Chlordane - not otherwise specified	delta-BHC
Dieldrin	Endosulfan I	Endosulfan II
Endosulfan sulfate	Endrin	Endrin aldehyde
Endrin ketone	gamma-BHC (Lindane)	gamma-Chlordane
Heptachlor	Heptachlor epoxide	Methoxychlor
Toxaphene		
8082		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
8260B		
1,1,1,2-Tetrachloroethane	1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene
1,3-Dichloropropane	1,4-Dichlorobenzene	2,2-Dichloropropane
2-Butanone (Methyl ethyl ketone, MEK)	2-Chloroethyl vinyl ether	2-Chlorotoluene
2-Hexanone	4-Chlorotoluene	4-Methyl-2-pentanone (Methyl isobutyl ketone

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Hazardous and Solid Waste, Organic	*8260B*	Acetone
Benzene	Bromobenzene	Bromochloromethane
Bromodichloromethane	Bromoform	Bromomethane
Carbon disulfide	Carbon tetrachloride	Chlorobenzene
Chlorodibromomethane (Dibromochloromethan	Chloroethane	Chloroform
Chloromethane	cis-1,2-Dichloroethene	cis-1,3-Dichloropropene
Dibromomethane	Dichlorodifluoromethane	Dichloromethane (Methylene chloride)
Ethylbenzene	Hexachlorobutadiene	Isopropylbenzene
Methyl-t-butyl ether	m-Xylene	Naphthalene
n-Butylbenzene	n-Propylbenzene	o-Xylene
p-Isopropyltoluene	p-Xylene	sec-Butylbenzene
Styrene	tert-Butylbenzene	Tetrachloroethene
Tetrahydrofuran	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	Trichloroethene	Trichlorofluoromethane
Vinyl acetate	Vinyl chloride	Xylenes (total)
8270C		
1,2,4,5-Tetrachlorobenzene	1,2,4-Trichlorobenzene	1,2-Dichlorobenzene
1,2-Diphenylhydrazine	1,3-Dichlorobenzene	1,4-Dichlorobenzene
2,4,5-Trichlorophenol	2,4,6-Trichlorophenol	2,4-Dichlorophenol
2,4-Dimethylphenol	2,4-Dinitrophenol	2,4-Dinitrotoluene (2,4-DNT)
2,6-Dichlorophenol	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol (o-Cresol)
2-Naphthylamine	2-Nitroaniline	2-Nitrophenol
3,3'-Dichlorobenzidine	3-Methylphenol (m-Cresol)	3-Nitroaniline
4,6-Dinitro-2-methylphenol	4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol
4-Chloroaniline	4-Chlorophenyl phenyl ether	4-Methylphenol (p-Cresol)
4-Nitroaniline	4-Nitrophenol	Acenaphthene
Acenaphthylene	Acetophenone	Aniline
Anthracene	Benzidine	Benzo(a)anthracene
Benzo(a)pyrene	Benzo(b)fluoranthene	Benzo(g,h,i)perlyene
Benzo(k)fluoranthene	Benzoic acid	Benzyl alcohol
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether
Bis(2-ethylhexyl)phthalate	Butyl benzyl phthalate	Carbazole
Chrysene	Dibenz(a,h)anthracene	Dibenzofuran
Diethyl phthalate	Dimethyl phthalate	Di-n-butyl phthalate
Di-n-octyl phthalate	Diphenylamine	Fluoranthene
Fluorene	Hexachlorobenzene	Hexachlorobutadiene
Hexachlorocyclopentadiene	Hexachloroethane	Hexachloropropene
Indeno(1,2,3-cd) pyrene	Isophorone	Naphthalene
Nitrobenzene	N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine	N-Nitrosopyrrolidine	Pentachlorophenol
Phenanthrene	Phenol	Pyrene
Pyridine		
8310		
Acenaphthene	Acenaphthylene	Anthracene
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Chrysene

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Hazardous and Solid Waste, Organic	*8310*	Dibenz(a,h)anthracene
Fluoranthene	Fluorene	Indeno(1,2,3-cd) pyrene
Naphthalene	Phenanthrene	Pyrene
8330		
1,3,5-Trinitrobenzene (1,3,5-TBN)	1,3-Dinitrobenzene (1,3-DNB)	2,4,6-Trinitrotoluene (2,4,6-TNT)
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Amino-4,6-dinitrotoluene (2-Am-DNT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
m-Nitrotoluene (3-Nitrotoluene, 3-NT)	Nitrobenzene	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocin
o-Nitrotoluene (2-Nitrotoluene, 2-NT)	p-Nitrotoluene (4-Nitrotoluene, 4-NT)	

Supplement A: Inorganic Analysis by Wet Chemistry

This supplement describes the quality control (QC) requirements, procedures, and measurements utilized in performing inorganic analyses by a variety of wet chemistry techniques. These techniques are those other than colorimetric autoanalyzer (Supplement B). The QC activities presented here are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SA 1.0 Data Quality Objectives

The general laboratory DQOs are listed in CTL QAM Section 1.2. How each of the general DQO categories are assessed for inorganic analyses and what are the nominal QC acceptance criteria for associated QC specifications, are described in the remainder of this section. Tables SA1 and SA2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical method and/or in the project plan.

SA 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see CTL QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see CTL QAM Section 4.4).

SA 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing MS samples, LCSs, and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SA1 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SA 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of matrix spike (MS) samples, and replicate analyses of laboratory control samples (LCS). Results from these measurements are compared to the criteria listed in Tables SA1 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SA 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in CTL QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SA1 or the project-specific RLs.

Table SA1. DQOs for Inorganic Analyses of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (mg/L)	Completeness[c] (%)
Alkalinity	90-110	80-120	≤ 20	20	100
Ammonia	90-110	80-120	≤ 20	1	100
BOD, 5-Day	Not applicable	Not applicable	≤ 20	2	100
Chlorophyll-a	Not applicable	Not applicable	≤ 20	6000	100
Color	Not applicable	Not applicable	≤ 20	5	100
Conductivity	Not applicable	Not applicable	≤ 20	1 uhmos/cm	100
Fluoride	90-110	80-120	≤ 20	0.8	100
Oil and Grease	90-110	80-120	≤ 20	1	100
pH	Not applicable	Not applicable	≤ 20	0.1	100
Residue – TDS	Not applicable	Not applicable	≤ 20	1	100
Residue – TS	Not applicable	Not applicable	≤ 20	1	100
Residue – TSS	Not applicable	Not applicable	≤ 20	1	100
Residue – TVS	Not applicable	Not applicable	≤ 20	1	100
Residue – TVSS	Not applicable	Not applicable	≤ 20	1	100
Sulfide	90-110	80-120	≤ 20	1	100
Turbidity	Not applicable	Not applicable	≤ 20	1	100

LCS = laboratory control sample MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ 10X MDL.
b. Typical values listed. Exact values depend on specific method.
c. Typical values listed. Exact values depend on specific method, compound and project.

SA 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SA 1.6 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SA 2.0 Method Requirements

SA 2.1 Criteria for Standards and Materials

Primary standards are purchased from the quality sources (e.g., Solutions Plus, VWR). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards, for calibration and QC measurements, are prepared from primary standards. Detailed instructions for preparation of secondary standards are

provided in the applicable CTL analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in CTL QAM Section 3.2. Additional material must meet the minimum requirements outlined in approved methods.

SA 2.2 Criteria for Instruments

All analytical instrumentation must meet all the requirements for the analytical method to be conducted (see CTL QAM Section 3.2.1) and must be equipped with the appropriate manifolds and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Instruments operation must always be in accordance with manufacturers' and methods' instructions, and performance criteria specified in the methods must be met before analysis of any samples.

SA 2.3 Criteria for Analysis

Wet chemistry analyses performed by the CTL laboratory may include both sample preparation/extraction methods and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the more frequently utilized techniques and methods are listed in the remainder of this section.

SA 2.3.1 Calibrated Methods

All calibrated methods involve the measurement of an initial analytical response of a standard or series of standards, and relating the response(s) mathematically to the concentration of analyte present in the sample. Most methods require multiple-point calibrations to be used. Calibration curves are characterized by upper and lower limits, and data is not reported outside these boundaries unless suitable dilution or concentration of the sample has been performed. All such manipulations are recorded and appropriately utilized in calculations of final results. In general, curve regressions for inorganic chemistry calibrated methods are linear, although multi-order curves may have applicability to some methods. All calibrations require initial and periodic verification using known standards from sources independent of the calibration standards. Details of calibration for specific methods may be found in the SOPs.

SA 2.3.1.1 Ammonia

The potentiometric method of ammonia analysis utilizes an ammonia combination electrode. The ammonia electrode is a combination pH electrode immersed in a solution which is seperated from the sample by a glass permeable membrane. Ammonia deffuses across the membrane until equilibrium between the sample and the internal filled solution is obtained. The response of the pH electrode to changes in hydrogen ion activity is related to the ammonia concentration in a NERSTIAN manner.

SA 2.3.1.2 Biochemical Oxygen Demand (BOD, 5-Day)

The method consists of filling with sample, to overflowing, and storing in airtight 300-mL glass and incubating it at 20^0C +/- 1^0C for 5 days. Dissolved oxygen is measured initially and after incubation, and the BOD is computed from the difference between initial and final DO measurements. A purchased commercial seed preparation is used to provide a population of microorganisms to oxidize the

biodegradable organic matter in the sample. A solution of glucose-glutamic acid is used as a control standard.

SA 2.3.1.3 Chlorophyll-a

A known volume of a water sample is filtered through a glass fiber filter. Chlorophyll-a present in the sample will be retained, in addition to other suspended material. The filter is extracted using a 90% acetone solution to dissolve the chlorophyll-a. Measure and record absorbances of sample at 750, 664, 647, and 630 nm. Calculate the chlorophyll-a concentration in the extract with the trichromatic formula. The chlorophyll-a concentration in the extract is then used to calculate the concentration in the original sample in mg/m^3.

SA 2.3.1.4 Color

Cobalt-platinum (Co-Pt) color, end-on visual comparison of the sample in matched color-comparison tubes, is made against a prepared series of standards.

SA 2.3.1.7 Conductivity (Specific Conductance)

The ability of a sample aliquot to carry an electric current (conductivity) is measured using a commercially available conductivity meter equipped with an electrode and temperature compensation mechanism. The meter is calibrated against standard KCl solutions spanning the measurement range of the meter. A calibration verification standard is required for each range used which contains reportable data.

SA 2.3.1.8 Fluoride

Fluoride is determined potentiometrically using a fluoride electrode in conjunction with a standard electrode and a pH meter, having an expanded millivolt scale. The fluoride electrode consists of a lanthanum fluoride crystal across which a potential is developed by fluoride ions. The concentration is thus arrived at based on the relative potentials of the calibration curve.

SA 2.3.1.9 pH

The activity of the hydrogen ion (H^+) is determined by potentiometric measurement using a standard glass hydrogen electrode and reference electrode. The electromotive force produced in the glass electrode system varies linearly with pH, and measurement in a sample is compared against a standard curve of buffers with different pH values.

SA 2.3.1.10 Sulfide

Sulfide is determined by adding excess iodine to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfate to sulfur under acidic conditions. The excess iodine is back titrated with phenylarseine oxide to determine the relative concentration of sulfide in the sample.

SA 2.3.1.11 Turbidity

The method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the intensity of scattered light, the higher the turbidity. Formazin polymer is used as the primary standard. A HACH nephelometer equipped with several ranges for measurement is used. Each range is calibrated with a single standard. Standards may be prepared fresh, in-house, with each use, although some "permanent" standards are also available. Permanent standard concentrations are verified annually.

SA 2.3.2 Titrated Methods

Titration generally involves two chemical species which combine in a known ratio when both are present in solution. If the concentration of one species is known (titrant), the concentration of the other (analyte) can be determined by use of an endpoint indicator and addition of the titrant until the endpoint is detected. The volume of titrant used, together with the combination ratio of the species, allows the calculation of the analyte concentration.

SA 2.3.2.1 Alkalinity

Hydroxyl ions present in a sample as a result of dissociation or hydrolysis of solutes react with additions of standard acid. Titration of the sample aliquot with sulfuric acid of standardized concentration to a pre-selected pH using a standard pH electrode is performed.

SA 2.3.3 Gravimetric Methods

Gravimetric techniques employ a sample preparation which results in a solid residue that is or contains the analyte of interest. The residue is dried, desiccated, and weighed. The concentration of the analyte in the original sample is then calculated using the dried residue weight and the initial sample volume (to obtain mg/L) or initial sample wet weight (to obtain % of total solids).

SA 2.3.3.1 Solids (Residues)

The total solids fraction of a sample is determined by driving off the aqueous fraction of a pre-characterized sub-sample and weighing the residue that remains. Pre-characterization may involve either taking a initial "wet" weight for expression as TS%, or an initial volume measurement for expression as mg/L TS. Solid matter in waters, either suspended or dissolved, is measured through filtration followed by appropriate analysis of either the weight retained on the filter (total suspended solids or TSS), or the weight obtained after drying the filtrate (total dissolved solids or TDS). Total volatile solids (TVS) or total volatile suspended solids (TVSS) fractions may also be determined from either total solids (TS) or total suspended solids (TSS) by heating the appropriate residues at 500^0C, and quantifying the weight loss in the desired units.

SA 2.3.3.2 Oil and Grease

A separately collected sample is preserved to pH< 2 with sulfuric acid, and the entire sample is serially extracted three times with trichlorotrifluroethane in a separatory funnel. The extract is dried, solvent evaporated and the resulting residue is weighed. Results are expressed in mg/L, based on the intial sample volume.

SA 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in standard operating procedures and appropriate instrument maintenance manuals. The applicable Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as required. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SA 4.0 Instrument Calibration and Frequency

Specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the analytical SOP for each method.

General calibration requirements of inorganic analyses are summarized in Table SA3. As previously stated, calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Table SA3 and corrective actions if requirements are not satisfied are applicable to all calibrated analytical methods. An instrumental analysis batch includes all samples and QC from prep batches contained in an instrumental analytical run, as well as calibration and instruement performance monitoring QC as addressed in Table SA3.

Table SA3. General Calibration Requirements for Inorganic Calibrated Methods

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Three-point to five-point initial calibration plus blank (ICAL)	Initially and as needed	$r \geq 0.995$ for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	Second source, %R: 90-110% for all analytes	Remake and reanalyze ICV standard once, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After each ICV, prior to sample analysis	< MDL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or repeat ICAL.
Continuing calibration verification (CCV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R: 90-110% for all analytes	Remake and reanalyze CCV once, if still unacceptable investigate and correct problem, cannot proceed until valid CCV obtained or ICAL repeated.
Continuing calibration blank (CCB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	< MDL	Remake and reanalyze CCB once, if still unacceptable investigate and correct problem, cannot proceed until valid CCB obtained or ICAL repeated.
%R = percent recovery RSD = relative standard deviation MDL = method detection limit			

SA 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all inorganic analyses, as applicable. The QA Coordinators and Analytical Section Managers are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Analytical Section Managers are responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SA4. Analytical QC samples are associated with field samples through the use of both instrumental analysis and preparation batches. A preparation or "prep" batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A prep batch must not exceed 20 field samples and must also contain all applicable preparation associated QC samples described below. More specific criteria are provided in the analytical method SOPs.

SA 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be acceptably demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses of a known standard and achieve precision and accuracy equal to or better than those listed in the method. In the absence of method criteria, the general criteria listed in Table SA4 are used.

SA 5.2 Blanks

A method blank (MB) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The MB is analyzed at the beginning of every analytical run and prior to the analysis of any samples. MB results are acceptable if the concentrations of the target analyte are not higher than the highest of ether the method detection limit, or five percent of the regulatory limit, or five percent of the measured concentration in the associated sample(s). If any target analyte concentration in the MB exceeds the criteria, the source of contamination must be identified and eliminated. Analysis of samples cannot proceed until a compliant MB is obtained.

SA 5.3 Duplicates

A duplicate sample (DUP) or duplicate matrix spike sample (MSD) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD), must be within the in-house determined acceptance ranges listed or project specified limits.

If the QC criteria for duplicate sample or spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SA 5.4 Matrix Spikes

Spikes (MS) are prepared every 20 field samples for each matrix, depending on the specific method or project requirements. Spike recoveries must fall within the in-house determined acceptance ranges listed or project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SA 5.5 Laboratory Control Samples

A laboratory control sample (LCS) is prepared at a frequency of one per every 20 field samples depending on the specific method or project requirements. The LCS results are acceptable if the percent recovery of each analyte is within the in-house determined acceptance range listed or project specified limits. If the LCS results do not meet specification, sample analyses must be stopped until the problem is corrected, and all associated samples in the analysis batch must be reanalyzed.

SA 5.6 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results is received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem. All findings must be documented and available for review.

Table SA4. Summary of QC Requirements for Inorganic Analyses

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	Method criteria for accuracy and precision, default ±20%	Retrain and repeat until acceptable
Method Blank (MB)	One (1) per analytical batch	< MDL, or 5% of the concentration in the associated samples, or 5% of the regulatory limit (highest of)	Halt analysis, take corrective action as needed to obtain acceptable criteria and reanalyze MB.
Laboratory control sample (LCS)	One (1) per analytical batch	In-house derived limits or 90% $\leq$ %R $\leq$ 110%, which ever is tighter or applicable	Halt analysis, take corrective action as needed to obtain acceptable criteria, reanalyze LCS.
Sample duplicate (DUP) or matrix spike duplicate (MSD)	One (1) per analytical batch	In-house derived limits or RPD $\leq$ 20% if analytes > MDL, which ever is tighter or applicable	Investigate problem, if LCS is in control the problem is judged to be matrix or solution related
Matrix spike sample (MS)	One (1) per analytical batch	In-house derived limits or 80% $\leq$ %R $\leq$ 120%, which ever is tighter or applicable	Investigate problem, if LCS is in control the problem is judged to be matrix or solution related.
Blind performance evaluation sample (PE)	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.

%R = percent recovery
MDL = method detection limit
RPD = relative percent difference

SA 6.0 Data Management

SA 6.1 Data Generation

Sample analyses at the laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see SB5.0).

SA 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis.

Deviations from the specified data reduction procedures are permitted only with approval of the applicable Analytical Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SA 6.3 Data Validation

As stated in QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary.
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is and archived in the associated data package/file.

SA 6.4 Data Reporting

After peer review of the data is completed, and the results approved, a report is generated. The applicable Project Manager (PM) reviews the report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections. The final report is signed by the PM before it is submitted to the customer. Each final report has a unique identification number, the CTL Folder No., listed in the upper right hand corner of the report.

The laboratory offers four levels of data reports (illustrated in QAM Table 7-1). For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide a copy of the analysis data and any related narrative comments to Project Management, where the completed package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of 5 years, or longer if requested by the client. These records are stored in the laboratory facility for about one year then transferred to another building for secure, long term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. The LIMS is maintained on-site by CTL's Information System personnel. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement B: Inorganic Analysis by Colorimetric Autoanalyzer

This supplement describes the quality control (QC) requirements, procedures, and measurements utilized in performing colorimetric analyses by autoanalyzers, specifically the Lachat QuikChem 4 and AE. These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SB 1.0 Data Quality Objectives

The general laboratory DQOs are listed in CTL QAM Section 1.2. How each of the general DQO categories are assessed for inorganic analyses by autoanalyzer, and what are the nominal QC acceptance criteria for associated QC specifications, are described in the remainder of this section. Tables SB1 and SB2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical method and/or in the project plan.

SB 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see CTL QAM Section 3.2.1), and completing the analysis within the applicable analysis holding time (see CTL QAM Section 4.4).

SB 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing MS samples, LCSs, and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SB1 and SB2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SB 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of matrix spike (MS) samples, and replicate analyses of laboratory control samples (LCS). Results from these measurements are compared to the criteria listed in Tables SB1 and SB2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Inorganic Analysis by Colorimetric Autoanalyzer

SB 1.0 Data Quality Objectives

SB 2.0 Method Requirements

SB 3.0 Instrument Testing, Inspection and Maintenance Requirements

SB 4.0 Instrument Calibration and Frequency

SB 5.0 Quality Control

SB 6.0 Data Management

SB 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in CTL QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SB1 and SB2 or the project-specific MDLs.

Table SB 1. DQOs for Inorganic Analyses by Autoanalyzer of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (mg/L)	Completeness[c] (%)
Alkalinity, as $CaCO_3$	90-110	80-120	$\leq$ 20	20	100
Ammonia, as N	90-110	80-120	$\leq$ 20	0.1	100
Chloride	90-110	80-120	$\leq$ 20	0.5	100
Chromium (Hexavalent)	90-110	80-120	$\leq$ 20	12	100
Cyanide	90-110	80-120	$\leq$ 20	0.005	100
Hardness, as $CaCO_3$	90-110	80-120	$\leq$ 20	20	100
Nitrate	90-110	80-120	$\leq$ 20	2	100
Nitrite	90-110	80-120	$\leq$ 20	0.2	100
Phenolics	60-140	60-140	$\leq$ 40	0.005	100
Phosphorus	90-110	80-120	$\leq$ 20	0.1	100
Sulfate	90-110	80-120	$\leq$ 20	1	100
Total Kjeldahl Nitrogen	90-110	80-120	$\leq$ 20	0.2	100

LCS = laboratory control sample MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations $\geq$ 10X MDL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound and project.

Table SB 2. DQOs for Inorganic Analyses by Autoanalyzer of Solid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (mg/kg)	Completeness$_c$ (%)
Ammonia, as N	90-110	80-120	≤ 20	1	100
Cyanide	90-110	80-120	≤ 20	0.7	100
Chromium (Hexavalent)	90-110	80-120	≤ 20	12	100
Nitrate	90-110	80-120	≤ 20	20	100
Nitrite	90-110	80-120	≤ 20	2	100
Phenolics	60-140	60-140	≤ 40	0.25	100
Sulfate	90-110	80-120	≤ 20	10	100
Total Phosphorus (non-Ortho)	90-110	80-120	≤ 20	0.7	100
Total Kjeldal Nitrogen	90-110	80-120	≤ 20	20	100

LCS = laboratory control sample MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation % R = percent recovery
a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ 10X MDL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound and project.

SB 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SB 1.6 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SB 2.0 Method Requirements

SB 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g., ERA). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in CTL QAM Section 3.2. Additional material must meet the minimum requirements outlined in approved methods.

SB 2.2 Criteria for Instruments

The colorimetric autoanalyzer instrument must meet all the requirements for the analytical method to be conducted (see CTL QAM Section 3.2.1) and must be equipped with the appropriate manifolds and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Instruments operation must always be in accordance with manufacturers' and methods' instructions, and performance criteria specified in the methods must be met before analysis of any samples.

SB 2.3 Criteria for Analysis

Colorimetric autoanalyzer (Lachat) analyses performed by the CTL laboratory include sample preparation/extraction and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section.

SB 2.3.1 Alkalinity

Methyl orange is used as a color indicator for determining alkalinity colorimetrically. Use of this indicator allows comparability to the equivalence point for alkalinity determined by titration. The methyl orange indicator is prepared in a dilute pH 3.1 buffer, which is just below its color change pH. When an alkaline sample is injected, the poorly buffered methyl orange changes color in proportion to the change in pH of the weak buffer, and thus proportional to the alkalinity of the sample, when measured at 550 nm.

SB 2.3.2 Ammonia

Based on the Berthelot reaction, ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity.The absorbance of the reaction product is directly proportional to the original ammonia concentration in the sample, when measured at 630 nm.

SB 2.3.3 Chloride

Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms the highly colored ferric thiocyanate, of which the absorbance, 480 nm, is proportional to the chloride concentration.

SB 2.3.4 Chromium (Hexavalient)

Hexavalent chromium is determined colormetrically by reaction with diphenylcarbizide in acidic solution, producing a red-violet color. The absorbance of the reaction product is directly proportional to the in the sample, when measured at 540 nm.

SB 2.3.5 Cyanide

Cyanide in the form of hydrocyanic acid (HCN) is released from cyanide complexes in samples by way of a manual reflux-distillation. The gaseous HCN produced is absorbed in a scrubber containing sodium hydroxide solution. The CN^- ion in the trapping solution

is converted to cyanogen chloride through reaction with Chloramine-T. This subsequently reacts with pyridine and barbituric acid to give a red-blue color complex, which absorbs at 570 nm.

SB 2.3.6 Hardness

Disodium magnesium EDTA is used to exchange magnesium on an equivalent basis for calcium and/or any other cation which forms a more stable EDTA chelate than magnesium. Non-chelated magnesium is then reacted with calmagite at a pH of 10.0 to form a red-violet complex with a maximum absorbance at 520 nm.

SB 2.3.7 Nitrate and/or Nitrite

Nitrate is reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide, followed by coupling with N-(1-naphyl)ethylenediamine dichloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrate alone can be determined by the same analysis, without the cadmium reduction column. Both analysis run in combination will the produce information for nitrite calculated concentration.

SB 2.3.8 Phenolics

Volatile phenolic compounds are separated from the sample matrix by distillation. The distillate is collected and analyzed colorimetrically, at 500 nm. Phenol, ortho- and meta-substituted phenols, and para-substituted phenols, where the para-group is a carboxyl, halogen, methoxyl, or sulfuric acid group, are all determined by a reaction with 4-aminoantipyrine. Para-cresol, and para-substituted phenols where the group is an alkyl, aryl nitro, benzoyl, nitroso, or aldehyde group are *not* determined.

SB 2.3.9 Phosphorus

Conversion of polyphosphates and organic phosphorus to orthophosphate is done through sulfuric acid and persulfate digestion. Existing orthophosphate can be measured without digestion. Orthophosphate ion (PO_4^{3-}) reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is directly proportional to the concentration of orthophosphate, and thus total converted phosphorus, in the sample.

SB 2.3.10 Sulfate

Interfering multivalent metal ions are removed with the use of a sodium form cation exchange column. The remaining sulfate is then reacted with an alcohol solution of barium chloride and methylthymol blue, at a pH of 2.5 – 3, to form barium sulfate. The combined solution is then raised to pH 12.5 – 13 so that excessive barium reacts with methylthymol blue, and measured at 460 nm. Initially, the barium and methylthymol blue are equimolor, equivalent to 300 mg sulfate / L, thus the amount of uncomplexed methylthymol blue is equal to the sulfate present.

SB 2.3.11 Total Kjeldahl Nitrogen

The preparatory digestion converts nitrogen components of biological origin (amino acids, proteins, peptides) to ammonia. It may *no*t convert the nitrogenous compounds of som industrial wastes (amines, nitor compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines). Ammonia is then measured colorimetrically through formation of indophenol blue (SB 2.3.2)

SB 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in standard operating procedures and appropriate instrument maintenance manuals. The applicable Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SB 4.0 Instrument Calibration and Frequency

Specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP for each method.

General calibration requirements of inorganic analyses by Lachat are summarized in Table SB3. As previously stated, calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Table SB3 and corrective actions if requirements are not satisfied are applicable to all autoanalyzer analytical methods.

SB 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all autoanalyzer analyses. The QA Coordinators and Analytical Section Managers are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Analytical Section Managers are responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SB4. Analytical QC samples are associated with field samples through the use of analysis batches. An analysis batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. An analysis batch must not exceed 20 field samples. More specific criteria are provided in the analytical method SOPs ..

Table SB 3. Autoanalyzer (Lachat) Calibration Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Three-point or five-point initial calibration plus blank (ICAL)	Initially and as needed	$\leq$ 20% RSD for individual response factors or r $\geq$0.99 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	Second Source, %R 90-110% for all analytes	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	< MDL	Remake and reanalyze ICB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or repeat ICAL
Calibration verification (CV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 90-110% for all analytes	Remake and reanalyze CV once, if still unacceptable investigate and correct problem, cannot proceed until valid CV obtained or ICAL repeated
Continuing calibration blank (CCB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	< MDL	Remake and reanalyze ICB once, if still unacceptable investigate and correct problem, cannot proceed until valid CCB obtained or repeat ICAL
%R = percent recovery RSD = relative standard deviation MDL = method detection limit			

SB 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be acceptably demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses and achieve precision and accuracy equal to or better than those listed in the method. In the absence of in-house determined criteria, the general criteria listed in Table SB4 are used.

SB 5.2 Blanks

A method blank (MB) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The MB is analyzed at the beginning of every analytical run and prior to the analysis of any samples. MB results are acceptable if the concentrations of the target analyte are not higher than the highest of either the method detection level, or five percent of the regulatory level, or five percent of the measured concentration in the associated sample(s). If any target analyte concentration in the MB exceeds the criteria, the source of contamination must be identified and eliminated. Analysis of samples cannot proceed until a compliant MB is obtained.

SB 5.3 Duplicates

A duplicate sample (DUP) or duplicate matrix spike sample (MSD) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD), must be within the in-house determined acceptance ranges or project specified limits.

If the QC criteria for duplicate sample or spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SB 5.5 Matrix Spikes

Spikes (MS) are prepared every 20 field samples depending on the specific method or project requirements. Spike recoveries must fall within the in-house determined acceptance ranges or project specified limits.If the QC criteria for the matrix spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SB 5.6 Laboratory Control Samples

A laboratory control sample (LCS) is prepared at a frequency of one per every 20 field samples depending on the specific method or project requirements. The LCS results are acceptable if the percent recovery of each analyte is within the in-house determined acceptance range or project specified limits. If the LCS results do not meet specification, sample analyses must be stopped until the problem is corrected, and all associated samples in the analysis batch must then be reanalyzed.

SB 5.7 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results is received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem. All findings must be documented and available for review.

Table SB 4. Summary of QC Requirements for Autoanalyzer Analysis

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	Method criteria for accuracy and precision, default ±20%	Retrain and repeat until acceptable.
Method blank (MB)	Daily, prior to sample analysis	< MDL, or 5% of the concentration in the associated samples, or 5% of the regulatory limit (highest of)	Halt analysis, take corrective action as needed to obtain acceptable criteria and reanalyze MB.
Laboratory control sample (LCS)	One (1) per analytical batch	In-house derived limits or 90% ≤ %R ≤ 110%, which ever is tighter or applicable	Halt analysis, take corrective action as needed to obtain acceptable criteria, reanalyze LCS.
Sample duplicate (DUP) or matrix spike duplicate (MSD)	One (1) per analytical batch	In-house derived limits or RPD ≤ 20% if analytes > MDL, which ever is tighter or applicable	Investigate problem, if LCS is in control the problem is judged to be matrix or solution related.
Matrix spike sample (MS)	One (1) per analytical batch	In-house derived limits or 80% ≤ %R ≤ 120%, which ever is tighter or applicable	Investigate problem, if LCS is in control the problem is judged to be matrix or solution related.
Blind performance evaluation sample	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.
%R = percent recovery RPD = relative percent difference MDL = method detection level			

SB 6.0 Data Management

SB 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see SB5.0).

SB 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis

Deviations from the specified data reduction procedures are permitted only with approval of the applicable Analytical Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SB 6.3 Data Validation

As stated in CTL QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary.
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented and archived in the associated data package/file.

SB 6.4 Data Reporting

After peer review of the data is completed, and the results approved, a report is generated. The applicable Project Manager (PM) reviews the report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections. The final report is signed by the PM before it is submitted to the customer. Each final report has a unique identification number, the CTL Folder No., listed in the upper right hand corner of the report.

The laboratory offers four levels of data reports (illustrated in QAM Table 7-1). For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide a copy of the analysis data and any related narrative comments to Project Management, where the completed package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of 5 years, or longer if requested by the client. These records are stored in the laboratory facility for about one year then transferred to another building for secure, long term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. The LIMS is maintained on-site by CTL's Information System personnel. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement C: Inorganic Analysis by Ion Chromatography

This supplement describes the quality control (QC) requirements, procedures, and measurements utilized in performing analysis for inorganic anions by ion chromatography. These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SC 1.0 Data Quality Objectives

The general laboratory DQOs are listed in CTL QAM Section 1.2. How each of the general DQO categories are assessed for inorganic analyses by ion chromatography, and what are the nominal QC acceptance criteria for associated QC specifications, are described in the remainder of this section. Tables SC1 and SC2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical and/or in the project plan.

SC 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see CTL QAM Section 3.2.1), and completing the analysis within the applicable analysis holding time (see CTL QAM Section 4.4).

SC 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing MS samples, LCSs, and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SC1 and SC2 or, if more specific criteria apply, to that listed in the applicable method's DVC. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SC 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of matrix spike (MS) samples, and replicate analyses of laboratory control samples (LCS). Results from these measurements are compared to the criteria listed in Tables SC1 and SC2 or, if more specific criteria apply, to the criteria in the applicable method's data validation checklist (DVC). These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SC 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in CTL QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SC1 and SC2 or the project-specific MDLs.

Table SC1. DQOs for Inorganic Analyses by Ion Chromatography of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (mg/L)	RL[c] (mg/L)	Completeness (%)
Bromide	90-110	80-120	$\leq$ 20	0.1	30	100
Chloride	90-110	80-120	$\leq$ 20	0.2	10	100
Fluoride	90-110	80-120	$\leq$ 20	0.2	0.8	100
Nitrate as N	90-110	80-120	$\leq$ 20	0.01	2	100
Nitrite as N	90-110	80-120	$\leq$ 20	0.01	0.2	100
Ortho-Phosphate as P	90-110	80-120	$\leq$ 20	0.5	1	100
Sulfate	90-110	80-120	$\leq$ 20	0.5	1	100

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations $\geq$ RL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound, and project.

LCS = laboratory control sample
% R = percent recovery
MS = matrix spike
% RSD = percent relative standard deviation
RPD = relative percent difference
MDL = method detection limit
RL = reporting limit

Table SC2. DQOs for Inorganic Analyses by Ion Chromatography of Solid Sample Extracts

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (mg/kg)	RL[c] (mg/kg)	Completeness (%)
Chloride	90-110	80-120	≤ 20	2	200	100
Fluoride	90-110	80-120	≤ 20	2	5	100
Nitrate as N	90-110	80-120	≤ 20	0.1	1	100
Nitrite as N	90-110	80-120	≤ 20	0.1	1	100
Ortho-Phosphate as P	90-110	80-120	≤ 20	5	10	100
Sulfate	90-110	80-120	≤ 20	5	50	100

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound, and project.

LCS = laboratory control sample
% R = percent recovery
MS = matrix spike
% RSD = percent relative standard deviation
RPD = relative percent difference
MDL = method detection limit
RL = reporting limit

SC 1.5 Quantitation/Reporting Limits

The capability to quantitate analytes at or below the limit of quantitation (LOQ) and/or reporting limit (RL) concentrations listed in Tables SC1 and SC2, or project-specified limits, is demonstrated by annual determination or verification of MDLs and by setting the concentration of at least one calibration standard at or below the LOQ/RL for each analyte.

SC 1.6 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SC 1.7 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SC 2.0 Method Requirements

SC 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g. Fisher Scientific). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in CTL QAM Section 3.2. Other materials used must meet the minimum requirements outlined in the approved methodology.

SC 2.2 Criteria for Instruments

Ion chromatographic instruments must meet all the requirements for the analytical method to be conducted (see CTL QAM Section 3.2.1) and must be equipped with the appropriate columns, supressors, and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Instruments operation must always be in accordance with manufacturers' and methods' instructions, and performance criteria specified in the methods must be met before analysis of any samples.

SC 2.3 Criteria for Analysis

Ion chromatography analyses performed by the CTL laboratory include sample preparation/extraction and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized follow in the remainder of this section.

A small volume of sample, typically 5 mL, is introduced into the ion chromatograph (Dionex DX-120) using an autosampler. The anions of interest (bromide, chloride, fluoride, nitrate, nitrite, *ortho*-phosphate and sulfate) are separated and measured using a system comprised of a guard column, analytical separation column, suppression device, and a conductivity detector. The mobile phase used is an eluent solution with a concentration of 1 mM sodium bicarbonate and 3.2 mM sodium carbonate in reagent water. The pump flow rate for the system is 2.00 mL/min.

Anion measurements in soils and solids involves a water extraction using five grams of the solid sample mixed with 50 mL of reagent water. The sample/water slurry is mixed for thirty minutes, then filtered through a 0.45 um membrane filter, and analyzed.

SC 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The applicable Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SC 4.0 Instrument Calibration and Frequency

Specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP and data validation checklist for each method.

General calibration requirements of inorganic analyses by ion chromatography are summarized in Table SC3. As previously stated, calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Table SC3 and corrective actions if requirements are not satisfied are applicable to all ion chromatography analytical methods.

SC 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all ion chromatography analyses. The QA Coordinators and Analytical Section Managers are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Analytical Section Managers are responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SC4. Analytical QC samples are associated with field samples through the use of preparation or "prep" batches. A prep batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A prep batch must not exceed 20 field samples. More specific criteria are provided in the analytical method SOPs and data validation checklists.

Table SC3. Ion Chromatograph Calibration Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Three-point or five-point initial calibration plus blank (ICAL)	Initially and as needed	$\leq$ 20% RSD for individual response factors or r$\geq$0.99 for regression line	Repeat until acceptable
Calibration Check Standard	After each ICAL, prior to sample analysis	%R 90-110% for all analytes	Remake and reaanalyze calibration check standard once, if still unacceptable repeat ICAL
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	Second source material, %R 90-110% for all analytes	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	< RL	Remake and reaanalyze ICB standard once, if still unacceptable repeat ICAL
Calibration verification (CV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 90-110% for all analytes	Remake and reanalyze CV once, if still unacceptable investigate and correct problem, cannot proceed until valid CV obtained or ICAL repeated
Calibration Blank (CB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	< RL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or ICAL repeated

%R = percent recovery
RSD = relative standard deviation

SC 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be acceptably demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses of a known standard and achieve precision and accuracy equal to or better than the most recent in-house determined acceptance ranges for laboratory duplicates and laboratory control samples, respectively. In the absence of in-house determined criteria, the general criteria listed in Table SC4 are used.

SC 5.2 Blanks

A method blank (MB) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The MB is analyzed at the beginning of every analytical run and prior to the analysis of any samples. MB results are acceptable if the concentrations of the target analyte does not exceed the reporting limit (RL). If any target analyte concentration in the MB exceeds the RL, the source of contamination must be identified and eliminated. Analysis of samples cannot proceed until a compliant MB is obtained.

SC 5.3 Duplicates

A duplicate sample (DUP) or duplicate matrix spike sample (MSD) is prepared at a frequency of one per 20 field samples depending on the specific method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD), must be within the in-house determined acceptance ranges listed in the data validation checklist or project specified limits.

If the QC criteria for duplicate sample or spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

Table SC4. Summary of QC Requirements for Ion Chromatography Analysis

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	In-house determined criteria for LCS recovery and duplicate precision	Repeat until acceptable
Method blank (MB)	Daily prior to sample analysis	Analytes < RL	Clean analytical system, repeat until MBs are in control
Sample duplicate (DUP) or matrix spike duplicate (MSD)	One (1) per analytical batch	In-house derived limits Default: RPD $\leq$ 20% if analytes > RL	Investigate problem, if system precision in control qualify results, if system precision out of control reanalyze entire batch
Matrix spike sample (MS)	One (1) per analytical batch	In-house derived limits Default: 80% $\leq$ %R $\leq$120%	Investigate problem, if system accuracy in control qualify results, if system accuracy out of control reanalyze entire batch
Laboratory control sample (LCS)	One (1) per analytical batch	Second source material, 90% $\leq$ %R $\leq$ 110%	Halt analysis, fix problem, repeat associated sample analyses
Blind performance evaluation sample (PE)	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results

%R = percent recovery RPD = relative percent difference
RL = reporting limit

SC 5.5 Matrix Spikes

Spikes (MS) are prepared every 20 field samples depending on the specific method or project requirements. Spike recoveries must fall within the in-house determined acceptance ranges listed in the data validation checklist or project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause of the problem must be determined and corrected. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SC 5.6 Laboratory Control Samples

A laboratory control sample (LCS) is second-source to the calibration standards and must be analyzed at a frequency of one per every 20 field samples depending on the specific method or project requirements. The LCS results are acceptable if the percent recovery of each analyte is within the in-house determined acceptance range listed in the data validation checklist or project specified limits. If the LCS results do not meet specification, sample analyses must be stopped until the problem is corrected, and all associated samples in the analysis batch must then be reanalyzed.

SC 5.7 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results is received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem. All findings must be documented and available for review.

SC 6.0 Data Management

SC 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see SB5.0).

SC 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis. The analyst then enters the results for both samples and QC measurements into the laboratory information management system (LIMS).

Deviations from the specified data reduction procedures are permitted only with approval of the applicable Analytical Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SC 6.3 Data Validation

As stated in CTL QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary, and completed data validation checklist
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented with a signed data validation checklist. The checklist is archived in the associated data package/file.

SC 6.4 Data Reporting

After peer review of the data is completed as described in Section SC6.3 and the results are approved, the analyst approves the result in LIMS and a report is generated. The applicable Project Manager (PM) reviews the report (CTL QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections. The final report (CTL QAM Exhibit 7-2) is signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Lab No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports (illustrated in QAM Table 7-1). For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 10 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. The LIMS is maintained on-site by CTL's Information System personnel. Full server backups are performed nightly. Other electronic data including instrument magnetic tape media are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement D: Metals Analysis

This supplement to the CTL's Quality Assurance Program Plan (QAM) describes the quality control (QC) requirements, procedures, and measurements utilized in performing metals analyses by cold vapor atomic absorption (CVAA), graphite furnace atomic absorption (GFAA), and inductivety coupled plasma (ICP). These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

Metals Analysis

SD 1.0 Data Quality Objectives

SD 2.0 Method Requirements

SD 3.0 Instrument Testing, Inspection and Maintenance Requirements

SD 4.0 Instrument Calibration and Frequency

SD 5.0 Quality Control

SD 6.0 Data Management

SD 1.0 Data Quality Objectives

The general laboratory DQOs are listed in Section 1.2 of the CTL QAM. How each of the general DQO categories are assessed for metals analyses by CVAA, GFAA, and ICP and what are the nominal QC acceptance criteria for associated QC specifications are described in the remainder of this section. Tables SD-1 and SD-2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical method and/or in the project plan.

SD 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see QAM Section 4.4).

SD 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing matrix spike (MS) samples, laboratory control samples (LCSs), and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SD-1 and SD-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SD 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of MS samples, and replicate analyses of LCSs. Results from these measurements are compared to the criteria listed in Tables SD-1 and SD-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Table SD-1. DQOs for Metals Analyses of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/L)	Completeness (%)
Mercury by CVAA	80-120	75-125	≤20	0.2	100
Metals by FLAA					
Potassium	80-120	75-125	≤20	10	100
Sodium	80-120	75-125	≤20	30	100
Metals by GFAA					
Antimony	80-120	75-125	≤20	3	100
Arsenic	80-120	75-125	≤20	1	100
Beryllium	80-120	75-125	≤20	0.2	100
Cadmium	80-120	75-125	≤20	0.1	100
Chromium	80-120	75-125	≤20	1	100
Copper	80-120	75-125	≤20	1	100
Lead	80-120	75-125	≤20	1	100
Selenium	80-120	75-125	≤20	0.6	100
Silver	80-120	75-125	≤20	0.2	100
Thallium	80-120	75-125	≤20	1	100
Metals by ICP					
Aluminum	80-120	75-125	≤20	20	100
Antimony	80-120	75-125	≤20	8	100
Arsenic	80-120	75-125	≤20	8	100
Barium	80-120	75-125	≤20	1	100
Beryllium	80-120	75-125	≤20	0.3	100
Boron	80-120	75-125	≤20	3	100
Cadmium	80-120	75-125	≤20	1	100
Calcium	80-120	75-125	≤20	10	100
Chromium	80-120	75-125	≤20	4	100
Cobalt	80-120	75-125	≤20	2	100
Copper	80-120	75-125	≤20	3	100
Iron	80-120	75-125	≤20	30	100
Lead	80-120	75-125	≤20	10	100
Magnesium	80-120	75-125	≤20	20	100
Manganese	80-120	75-125	≤20	1	100
Molybdenum	80-120	75-125	≤20	4	100
Nickel	80-120	75-125	≤20	5	100
Potassium	80-120	75-125	≤20	80	100
Selenium	80-120	75-125	≤20	20	100
Silver	80-120	75-125	≤20	2	100
Sodium	80-120	75-125	≤20	100	100
Thallium	80-120	75-125	≤20	20	100
Vanadium	80-120	75-125	≤20	3	100
Zinc	80-120	75-125	≤20	2	100

LCS = laboratory control sample MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

Table SD-2. DQOs for Metals Analyses of Solid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/kg)	Completeness (%)
Mercury by CVAA	80-120	75-125	≤20	2	100
Metals by FLAA					
Potassium	80-120	75-125	≤20	1000	100
Sodium	80-120	75-125	≤20	3000	100
Metals by GFAA					
Antimony	80-120	75-125	≤20	80	100
Arsenic	80-120	75-125	≤20	500	100
Beryllium	80-120	75-125	≤20	2	100
Cadmium	80-120	75-125	≤20	5	100
Chromium	80-120	75-125	≤20	100	100
Copper	80-120	75-125	≤20	700	100
Lead	80-120	75-125	≤20	700	100
Selenium	80-120	75-125	≤20	600	100
Silver	80-120	75-125	≤20	500	100
Thallium	80-120	75-125	≤20	700	100
Tin	80-120	75-125	≤20	700	100
Metals by ICP					
Aluminum	80-120	75-125	≤20	2000	100
Antimony	80-120	75-125	≤20	800	100
Arsenic	80-120	75-125	≤20	800	100
Barium	80-120	75-125	≤20	100	100
Beryllium	80-120	75-125	≤20	30	100
Boron	80-120	75-125	≤20	300	100
Cadmium	80-120	75-125	≤20	100	100
Calcium	80-120	75-125	≤20	1000	100
Chromium	80-120	75-125	≤20	400	100
Cobalt	80-120	75-125	≤20	200	100
Copper	80-120	75-125	≤20	300	100
Iron	80-120	75-125	≤20	3000	100
Lead	80-120	75-125	≤20	1000	100
Magnesium	80-120	75-125	≤20	2000	100
Manganese	80-120	75-125	≤20	100	100
Molybdenum	80-120	75-125	≤20	400	100
Nickel	80-120	75-125	≤20	500	100
Selenium	80-120	75-125	≤20	2000	100
Silver	80-120	75-125	≤20	200	100
Thallium	80-120	75-125	≤20	2000	100
Vanadium	80-120	75-125	≤20	300	100
Zinc	80-120	75-125	≤20	200	100

LCS = laboratory control sample MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery
a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations $\geq$ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

SD 1.4 Detectability

Method detection limits (MDLs) are determined or verified at least annually using the procedure and calculation described in QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SD-1 and SD-2 or the project-specific MDLs.

SD 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SD 1.6 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SD 2.0 Method Requirements

SD 2.1 Criteria for Standards and material

Primary standards are purchased from the best available source (e.g., CPI, Solutions Plus). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in QAM Section 3.2. Other material used in tests must meet the minimum requirements found in the approved methodology.

SD 2.2 Criteria for Instruments

Each instrument used for metals analyses must meet all the requirements for the analytical method (see QAM Section 3.2.1) and must be equipped with the appropriate samplers and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Instruments operation must always be in accordance with manufacturers' and methods' instructions, and instrument performance criteria specified in a method must be met before analysis of any samples.

SD 2.3 Criteria for Analysis

Metals analyses performed by the CTL laboratory include sample preparation/extraction methods and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section. All metals analysis are batched in groups of 20 or less samples of similar matricies

SD 2.3.1 Acid Digestion of Aqueous Samples and Extracts for ICP

A mixed acid digestion procedure is used to prepare water samples for metals analysis by inductively coupled plasma (ICP). Non-preserved water samples may be filtered through a 0.45 µm membrane filter, prior to preservation / digestion, if dissolved metal concentrations are to be determined. The samples are acid-digested with nitric acid and hydrochloric acid on a hot plate or in a block heater to digest the organic matter and dissolve the metals. Samples are monitored periodically during digestion to ensure an adequate, non-boiling reflux, and are not allowed to go to dryness. The digestates from this method can be analyzed for metals by ICP.

SD 2.3.2 Acid Digestion of Aqueous Samples and Extracts for GFAA

An acid digestion procedure is used to prepare water samples for graphite furnace atomic absorption (GFAA) metals analysis. Non-preserved water samples may be filtered through a 0.45 µm membrane filter, prior to preservation / digestion, if dissolved metal concentrations are to be determined. The samples are acid-digested with nitric acid (and hydrogen peroxide for As and Se) in a block heater to digest the organic matter and dissolve the metals. Samples are monitored periodically during digestion to ensure an adequate, non-boiling reflux, and are not allowed to go to dryness.

SD 2.3.3 Acid Digestion of Soils and Sediments for Metals by ICP, and GFAA

A mixed acid digestion procedure is used to prepare soil, sediment, and sludge samples for metals analysis. The samples are digested with nitric acid, hydrogen peroxide, and hydrochloric acid (for ICP) or nitric acid and hydrogen peroxide (for GFAA) in a block heater to remove the organic matter and dissolve the metals. Samples are monitored periodically during digestion to ensure an adequate, non-boiling reflux, and are not allowed to go to dryness. The digestate is then analyzed.

SD 2.3.4 TCLP Preparation for Metals

Toxicity characteristic leaching procedure (TCLP) is EPA SW-846 Method 1311 for determining the mobility of both organic and inorganic contaminants including metals present in all types of environmental matrices. TCLP leachates are prepared from soil and solid waste samples containing minimal or no filterable fluids. Sample leachates are generated using pH-adjusted acetic acid-based extraction fluids at amounts equal to 20 times the mass of the solid being leached. The specific extraction fluid employed is a function of the alkalinity of the solid phase of the sample. Following an 18 hour extraction, the TCLP extract is filtered, acid digested, and submitted for appropriate metals analyses.

SD 2.3.5 Acid Digestion and Analysis of Solid and Liquid Samples for Mercury by CVAA

Water samples are heat digested at 95°C in a water bath with a mixture of sulfuric acid, nitric acid and permanganate to oxidize organic materials and convert organo-mercurials to the mercuric ion. Persulfate oxidation is included to ensure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. Soil samples are heat digested at 95ºC in a water bath with a mixture of nitric acid, sulfuric acid, and permanganate (and persulfate for soils) to oxidize organic materials and convert organo-mercurials to the mercuric ion. Following digestion, excess permanganate is reduced with hydroxlamine hydrochloride. Mercury is measured by cold vapor atomic absorption (CVAA) analysis.

Mercury is reduced to the elemental state with stannous chloride solution and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of the CVAA. Mercury concentrations are measured as a function of absorbance.

SD 2.3.6 Metals Analysis by ICP

This technique determines metallic elements in digestates using inductively coupled plasma (ICP) atomic emission spectroscopy. The method is applicable to a large number of metals and samples types, all sample matrices require digestion prior to analysis. For dissolved analytes, all samples are preserved with nitric acid to a pH < 2 immediately following field filtration. The ICP emission spectrometer used allows simultaneous multiple metal determination of elements by measuring element specific light at precise wavelengths by optical spectrometry. Samples are nebulized, the resulting aerosol is transported to the plasma torch, and element-specific atomic-line emission spectra are measured electronically.

SD 2.3.7 Metals Analysis by GFAA

Graphite furnace atomic absorption (GFAA) spectrophotometry is used for the determination of metals at trace levels in all samples matrices. It involves injecting a fixed volume of digested sample into an electrically heated graphite furnace atomizer. A light beam from a hollow cathode lamp, specific to the element to be determined, is directed through the atomizer into a monochromator and absorption is measured, typically by dual beam optical systems. Absorption follows Beer's Law and is directly proportional to the concentration of the specific metal atoms in the light atomizer light path. For dissolved analytes, all samples are preserved with nitric acid to a pH < 2 immediately following field filtration.

SD 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The Metals Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SD 4.0 Instrument Calibration and Frequency

General calibration requirements for metals analyses by CVAA and GFAA are summarized in Table SD-3, and requirements for metals analyses by ICP are summarized in Table SD-4. Calibration requirements for specific reference methods or project may differ slightly from the listed general requirements, but the components of the calibration process described in Tables SD-3 and SD-4 and corrective actions if requirements are not satisfied are applicable to all metals analytical methods.

As previously stated, specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP and for each method.

SD 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all metals analyses. The QA Coordinators and Metals Section Manager are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Metals Section Manager is responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies by instrumental technique type are summarized in Tables SD-5 (CVAA and GFAA) and SD-6 (ICP). Analytical QC samples are associated with field samples through the use of preparation batches. A preparation batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A preparation batch must not exceed 20 samples. The acceptance criteria listed in Tables SD-5 and SD-6 are general guidance values only because the specific criteria depend on the individual metal, specific reference method, and any special project requirements. More specific criteria are provided in the analytical method SOPs.

Table SD-3. Calibration Requirements for Mercury by CVAA and GFAA

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Five-point initial calibration plus blank	Initially and as needed	r≥0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R 90-110% for all analytes	Reanalyze ICV standard once. If it is still unacceptable, repeat ICAL.
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	< MDL or RL	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Continuing Calibration Verification (CCV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 80-120% for all analytes	Reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until a valid CV is obtained or ICAL is repeated. Must reanalyze samples analyzed since last acceptable CV.
Continuing Calibration Blank (CCB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	< MDL or RL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or ICAL repeated.

%R = percent recovery
RSD = relative standard deviation
MDL or RL= method detection limit or reporting limit

Table SD-4. General Metals Instrument Calibration Requirements: ICP

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Linear dynamic range study	Annually	r≥0.995 for regression line	Repeat until acceptable
Two-point initial calibration plus blank	Initially and as needed	r≥0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R 90-110% for all analytes	Reanalyze ICV standard once. If it is still unacceptable, repeat ICAL.
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	±2x MDL or RL	Remake and reaanalyze ICB standard once, if still unacceptable repeat ICAL
Calibration verification (CV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 90-110% for all analytes	Reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until a valid CV is obtained or ICAL is repeated. Must reanalyze samples analyzed since last acceptable CV.
Calibration Blank (CB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	±2x MDL or RL	Remake and reanalyze CB twice, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or ICAL repeated.

%R = percent recovery
RSD = relative standard deviation
MDL or RL= method detection limit or reporting limit

Table SD-5. Summary of QC Requirements for Mercury Analysis by CVAA and GFAA

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration	Four (4) prepared samples analyzed prior to any customer sample analyses	Method criteria for LCS recovery and duplicate precision	Repeat until acceptable
Method blank	Daily prior to sample analysis	Analytes ≤ MDL or RL	Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. Reanalyze any samples > RL and <20x MB level.
Laboratory control sample	One (1) per preparation batch	Lab generated limits or defaults for Liquid: 80% ≤%R≤120% Solid: within vendor limits	Investigate and identify the problem. If system accuracy is in control (e.g., MS acceptable), no corrective action needed. If system is out of control, reanalyze entire batch.
Matrix spike sample	One (1) per preparation batch	Lab generated limits or default 75% ≤%R≤125% (Not applicable if sample is > 4X spike level)	Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch.
Sample duplicate or matrix spike duplicate	One (1) per preparation batch	RPD ≤20% if analyte > 5X RL	Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch.
Post digestion spike	As needed to confirm matrix effects	85%≤%R<115%	Repeat analysis. If it is still out, investigate for possible matrix effect or system problem.
Method of standard additions	As needed for samples with suspected or confirmed matrix effects	r≥0.995	Repeat analysis. If it is still out, investigate for possible matrix effect or system problem.
Blind performance evaluation sample	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.

PE = performance evaluation
QC = quality control
LCS = laboratory control sample
MB = method blank
MDL = method detection limit
%R = percent recovery
RL = reporting limit
RPD = relative percent difference

Table SD-6. Summary of QC Requirements for Metals Analysis by ICP

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration	Four (4) prepared samples analyzed prior to any customer sample analyses	Method criteria for LCS recovery and duplicate precision	Repeat until acceptable
Interference Check Samples (ICSA and ICSAB)	At the beginning of each analytical run, or twice per 8 hours,but not before the CCV	80% ≤%R≤120% for target analytes, <2X MDL or RL for non-target analytes	Recalibrate and reanalyze back to last acceptable ICSA / ICSAB
Method blank	Daily prior to sample analysis	Analyte ≤ MDL or RL	Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. Reanalyze any samples >RL and <20x MB level.
Laboratory control sample	One (1) per preparation batch	Liquid: 80% ≤%R≤120% Solid: within vendor limits	Investigate and identify the problem. If system accuracy is in control (e.g., MS acceptable), no corrective action needed. If system is out of control, reanalyze entire batch.
Matrix spike sample	One (1) per preparation batch	75% ≤%R≤125%	Investigate problem. If system accuracy is in control perform a PDS. If system accuracy is out of control, reanalyze entire batch.
Sample duplicate or matrix spike duplicate	One (1) per preparation batch	RPD ≤20% if analyte >5x RL	Investigate problem. If system precision is in control, perform a PDS. If system precision is out of control, reanalyze entire batch.
Post digestion spike (PDS)	As needed to confirm matrix effects	85%≤%R<115%	If PDS passes do not flag, if PDS fails flag data
Serial dilution	As needed to assess new and unusual matrices	RPD≤10%	Repeat analysis. If it is still out, investigate for possible matrix effect or system problem.
Method of standard additions	As needed for samples with suspected or confirmed matrix effects	r≥0.995	Repeat analysis. If it is still out, investigate for possible matrix effect or system problem.
Blind performance evaluation sample	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.

PE = performance evaluation
QC = quality control
LCS = laboratory control sample
MB = method blank
MDL = method detection limit
%R = percent recovery
RL = reporting limit
RPD = relative percent difference

SD 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses and achieve precision and accuracy within the acceptance ranges for laboratory duplicates and laboratory control samples, respectively. General acceptance criteria are listed in Tables SD-5 and SD-6.

SD 5.2 Blanks

A method blank (MB) is analyzed at the beginning of every analytical run and prior to instrumental analysis of any samples. MB results are acceptable if the concentrations of all target metals do not exceed reporting limits (RLs).

If any target metal concentration in the MB exceeds its RL, the source of contamination must be identified and eliminated. If contamination found in a MB is found in associated samples (where the sample level is >RL and <20x MB level), the samples should be re-digested. If it is not possible to re-digest the samples, the data must be appropriately qualified and the project manager notified. If contamination found in a method blank is not found in associated samples (where the sample level is <RL or $\geq$20x MB level) , the samples' results may be reported, but the source of the contamination should still be investigated and eliminated.

SD 5.3 Laboratory Control Samples

A laboratory control samples (LCS) is second-source to the calibration standards and must be analyzed at a frequency of one per every 10 to 20 samples depending on the specific reference method or project requirements. The LCS results are acceptable is the percent recovery of each analyte is $\geq$ 80% and $\leq$ 120% for liquids and within vendor provided acceptance range for solids or within project specified limits.

If the LCS results do not meet specifications, the cause and impact of the problem must be determined. If the LCS results appear anomalous (e.g., isolated digestion problem, bad injection into instrument) and the matrix spike results and other batch QC are acceptable, the samples' results for the batch are acceptable. If both the LCS and MS results are unacceptable, then instrumental analysis of the batch must be repeated. If after instrumental reanalysis both the LCS and MS are still unacceptable, then the batch must be re-digested. If it is not possible to re-digest, the project manager must be notified. The project manager will contact the client and possibly arrange for recollection of samples.

SD 5.4 Matrix Spikes

Spikes are run every 10 to 20 samples depending on the specific reference method or project requirements. Spike recoveries must be $\geq$75% and $\leq$125% or within project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause and impact of the problem must be determined. If the associated LCS results are acceptable, then the MS results are reportable with the qualifier that a matrix effect was observed. If the problem adversely affected the entire analysis batch, such as LCS results also unacceptable (see LCS discussion in SD 5.3), all samples in the batch must be re-digested and instrumentally reanalyzed.

SD 5.5 Duplicates

A duplicate sample or duplicate matrix spike sample is analyzed at a frequency of one per 10 to 20 samples depending on the specific method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than 10 times their respective MDL, or between a matrix spike (MS) and matrix spike duplicate (MSD) must be $\leq$20% or within project specified limits.
If the QC criteria for duplicate sample or spike analyses are not satisfied, the cause and impact of the problem must be determined. If the problem adversely affected the entire analysis batch, all samples in the batch must be re-digested and instrumentally reanalyzed.

SD 5.6 Post Digestion Spikes

Post digestion spike (PDS) is analyzed as needed to evaluate matrix effects. Generally, when a matrix spike recovery is outside the 75-125% acceptance range, a PDS is used to determine if the sample digestate matrix is interfering with the analysis of the analyte. The sample is spiked at a level consistent with the matrix spike. Acceptable recovery is within 15% of the spike true value, indicating that the sample digestate matrix is not interfering with analysis. If the recovery is outside of that range, than the digestate matrix is causing interference with analysis and the PDS may be used as the single addition method of standard additions (MSA) to correct the sample result and to recalculate the MDL, as appropriate.

SD 5.7 Serial Dilution

Serial dilution of sample digestate is performed as need to evaluate matrix effects. Generally, sample digestate is diluted 1:5 with method blank solution and analyzed. For diluted results $\geq$10x the MDL, the diluted digestate result should be within 10% of the undiluted digestate result, or a matrix effect is suspected. This is performed on the MS/MSD sample for each batch of solid samples or as required by a project.

SD 5.8 Method of Standard Additions

Method of standard additions (MSA) is used as needed to analyze samples with suspected or confirmed matrix effects (e.g., TCLP extracts). Equal volumes of sample are added to a water blank and three separate aliquots of standard containing approximately 50%, 100%, and 150% of the expected amount of metal. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as a linear regression of the absorbance plot should be linear (i.e., $r \geq 0.995$) or repeat MSA analysis once.

SD 5.9 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results are received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem.

SD 6.0 Data Management

SD 6.1 Data Generation

Qualified analysts perform sample analyses at the CTL laboratory, and appropriate analytical methods are used to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see Section SD 5.0).

SD 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis.

Deviations from the specified data reduction procedures are permitted only with approval of the Metals Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SD 6.3 Data Validation

As stated in QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results .
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented and archived in the associated data package/file.

SD 6.4 Data Reporting

After peer review of the data is completed as described in Section SD 6.3 and the results are approved, the analyst approves the results in LIMS and a preliminary report is generated. The applicable Project Manager (PM) reviews the preliminary report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections, then a final report is produced. The final report (QAM Exhibit 7-2) is also reviewed and signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Work Order No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports as illustrated in QAM Table 7-1. For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 3 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long-term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. CTL's Information System personnel maintain the LIMS on-site. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement E: Organic Analysis by Gas Chromatography

This supplement to the CTL's Quality Assurance Manual (QAM) describes the quality control (QC) requirements, procedures, and measurements utilized in performing organic analyses by gas chromatography (GC). These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SE 1.0 Data Quality Objectives

The general laboratory DQOs are listed in Section 1.2 of the CTL QAM. How each of the general DQO categories are assessed for organic analyses by GC and what are the nominal QC acceptance criteria for associated QC specifications are described in the remainder of this section. Tables SE-1 and SE-2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical method and/or in the project plan.

SE 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see QAM Section 4.4).

SE 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing matrix spike (MS) samples, laboratory control samples (LCSs), and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SE-1 and SE-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SE 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of MS samples, and replicate analyses of LCSs. Results from these measurements are compared to the criteria listed in Tables SE-1 and SE-2 or, if more specific criteria apply, to the criteria in the applicable method's DVC. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Organic Analysis by Gas Chromatography

SE 1.0 Data Quality Objectives

SE 2.0 Method Requirements

SE 3.0 Instrument Testing, Inspection and Maintenance Requirements

SE 4.0 Instrument Calibration and Frequency

SE 5.0 Quality Control

SE 6.0 Data Management

Table SE-1. DQOs for Organic Analyses by Gas Chromatography of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/L)	Completeness (%)
Pesticides / PCBs	70-130	70-130	≤ 20	0.5	100
Petroleum hydrocarbons	60-140	60-140	≤ 20	100	100
Volatile organic compounds	70-130	70-130	≤ 20	0.3	100

LCS = laboratory control sample
MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound, and project.

Table SE-2. DQOs for Organic Analyses by Gas Chromatography of Solid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/kg)	Completeness (%)
Pesticides / PCBs	70-130	70-130	≤ 20	0.5	100
Petroleum hydrocarbons	60-140	60-140	≤ 20	2000	100
Volatile organic compounds	70-130	70-130	≤ 20	25	100

LCS = laboratory control sample
MDL = method detection limit RPD = relative percent difference
MS = matrix spike % RSD = percent relative standard deviation
% R = percent recovery

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound.
c. Typical values listed. Exact values depend on specific method, compound, and project.

SE 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SE-1 and SE-2 or the project-specific MDLs.

SE 1.6 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SE 1.7 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SE 2.0 Method Requirements

SE 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g., Accu Standards, Absolute, Ultra Scientific, Chem Services). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in QAM Section 3.2. Other material used in testing must meet the minimum requirements identified in the approved methodology.

SE 2.2 Criteria for Instruments

Each gas chromatograph used for organic analyses must meet all the requirements for the analytical method (see QAM Section 3.2.1) and must be equipped with the appropriate separation columns and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Operation of instruments must always be in accordance with manufacturers' and methods' instructions, and instrument performance criteria specified in a method must be met before analysis of any samples.

SE 2.3 Criteria for Analysis

GC-based organic analyses performed by the CTL laboratory include sample preparation/extraction methods, clean-up methods, and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section.

SE 2.3.1 Liquid/Liquid Extraction-Separatory Funnel

Liquid/liquid extraction with separatory funnel is used to extract semivolatile organic compounds from liquid samples. The sample is pH-adjusted, if necessary, then extracted with methylene chloride in a separatory funnel. Residual water is removed from the extract with sodium sulfate, then the extract is concentrated by evaporation. For analysis of pesticides and PCBs, the extract is solvent exchanged into hexane.

SE 2.3.2 Pressurized Fluid Extraction

Pressurized Fluid Extraction (PFE) is a procedure for extracting water insoluble or water slightly soluble semi-volatile organic compounds from clays, sediments, sludges and waste solids. The method uses an elevated temperature and pressure to achieve analyte recoveries equivalent to those from soxhlet extraction, using less solvent and taking significantly less time than the soxhlet procedure.

SE 2.3.3 Solid-Phase Extraction

Solid-phase extraction (SPE) is used to extract semivolatile and other extractable organic compounds from aqueous samples. A measured volume of sample is adjusted to a specified pH and then extracted using a SPE device. Target analytes are eluted from the solid-phase media using methylene chloride or other appropriate solvent. Residual water is removed from the resulting solvent extract using sodium sulfate, then the extract is concentrated by evaporation. The concentrated extract may be exchanged into a solvent compatible with subsequent cleanup procedures or determinative procedures employed for the measurement of the target analytes.

SE 2.3.4 Ultrasonic Extraction

Ultrasonic extraction is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids, such as soils, sludges and solid wastes. A measured volume of sample is mixed with a drying agent to form a free flowing powder. This is solvent extracted three times using ultrasonic extraction. The resulting extract is ready for clean-up and/or analysis following concentration.

SE 2.3.5 Purge and Trap Extraction

Purge and trap extraction is used for extracting volatile organic compounds (VOCs) from water and soil samples, linked to analyses of VOCs by GC and GC/MS. The extraction is identical regardless of the measurement procedure. Helium is "purged" through the sample, stripping the volatiles and carrying them to a sorbent trap downstream. The analytes are thermally desorbed and backflushed into a GC where the components are separated and analyzed using a variety of detectors. Highly contaminated soil samples may be extracted with methanol prior to analysis. Highly contaminated water samples are often diluted prior to analysis.

SE 2.3.6 TCLP Preparation for Organics

Toxicity characteristic leaching procedure (TCLP) is EPA SW-846 Method 1311 for determining the mobility of both organic and inorganic contaminants present in all types of environmental matrices. TCLP leachates are prepared from soil and solid waste samples containing minimal or no filterable fluids. Sample leachates are generated using pH-adjusted acetic acid-based extraction fluids at amounts equal to 20 times the mass of the solid being leached. The specific extraction fluid employed is a function of the alkalinity of the solid phase of the sample. Following an 18 hour extraction, the TCLP extract is filtered and submitted for appropriate organics or metals analyses.

SE 2.3.7 TCLP ZHE Preparation for Organics

Toxicity characteristic leading procedure (TCLP) using a zero-headspace extraction vessel (ZHE) is part of EPA SW-846 Method 1311 for determining the mobility of volatile organic contaminants present in all types of environmental matrices. The ZHE device allows for liquid/solid separation, extraction, and final extract filtration without opening the vessel, so loss of volatiles is precluded. TCLP leachates are prepared from soil and solid waste samples containing minimal or no filterable fluids. Sample leachates are generated in a ZHE device using pH-adjusted acetic acid-based extraction fluid at amounts equal to 20 times the mass of

the solid being leached. Following the 18 hour extraction, the TCLP extract is filtered, stored with minimal headspace, and submitted for volatile organics analysis.

SE 2.3.8 Petroleum Hydrocarbons

Petroleum hydrocarbons including volatile and semivolatile are determined by a capillary GC technique with flame ionization detection (FID). Volatile products (e.g., gasoline) are determined using purge and trap (P/T) extraction, semivolatile products (e.g., diesel and lube oils) are determined on solvent extracts. Calibrations are conducted using products, typically, gasoline, diesel, jet fuels, or some type of residual fuel oil. All petroleum products should be referenced to a hydrogen specific range using synthetic n-alkyl hydrocarbons. Extractable hydrocarbons as diesel correspond to an approximate alkane range of C10-C28. Volatile hydrocarbons as gasoline typically correspond to alkane ranges between C4 and C8, and include the BTEX components (i.e., benzene, toluene, ethylbenzene, xylene).

P/T or solvent extracts are applied onto a capillary column in a GC equipped with FID. Quantification is performed by comparing the total chromatographic area of the petroleum product standard (e.g., diesel or gasoline, including resolved and unresolved components) to the response of the sample. Both volatile and extractable hydrocarbon determinations are typically required for full site characterization and closure.

SE 2.3.9 Volatile Organic Compounds by GC

This is a GC method to determine selected volatile organic compounds (VOCs) in a variety of matrices. This method is applicable to nearly all types of samples including, but not limited to, ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, soils, and sediments. The principal target compounds for which this method is utilized are the BTEX compounds (i.e., benzene, toluene, ethylbenzene, and xylene) and trichlorobenzene. Purge and trap extraction is used to isolate VOCs from an aliquot of liquid samples or from aqueous or methanol/aqueous extracts of solid or highly contaminated samples. Temperature programmed capillary column chromatography is used for compound separation and photoionization detector (PID) for compound detection. Qualitative identifications are attained by analyzing reference standards under the same conditions used for samples and by comparing resultant GC retention times.

SE 2.3.10 Selected Organic Compounds by Direct Injection GC

This is a capillary column GC technique using flame ionization detector (FID) for identifying and measuring selected organic compounds (e.g., glycols) from aqueous or soil samples. This technique is principally used for compounds that are very water soluble and are therefore not efficiently extracted by the purge and trap technique. Qualitative identifications are attained by analyzing reference standards under the same conditions used for samples and comparing resultant GC retention times.

SE 2.3.11 Pesticides / PCBs

This is a GC method to determine selected organochlorine pesticides and polychlorinated biphenyls (PCBs) in a variety of matrices. This method is applicable to nearly all types of samples including, but not limited to, ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, soils, and sediments. Sample extracts are injected into a gas chromatograph for seperation and detection of analytes of interest Temperature programmed

capillary column chromatography is used for compound separation and electron capture detector for compound detection. Qualitative identifications are attained by analyzing reference standards under the same conditions used for samples and by comparing resultant GC retention times.

SE 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The Organics/GC Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SE 4.0 Instrument Calibration and Frequency

General calibration requirements for organic analyses by GC are summarized in Table SE-3. Calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Table SE-3 and corrective actions if requirements are not satisfied are applicable to all GC analytical methods.

Table SE-3. GC Calibration Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Three-point or five-point initial calibration plus blank	Initially and as needed	Individual response factors for standards must exhibit either $\leq$20% RSD or r$\geq$0.99 for regression line.	Correct problem and repeat until acceptable.
Retention time windows	With every ICAL	RT windows must be centered on mid-concentration standard and established from three injections or other documented approach.	If analyte peaks are not within RT windows, reestablish RT windows or recalibrate.
Initial calibration verification	After each ICAL, prior to sample analysis	%R 85-115% for all analytes	Remake and reanalyze ICV standard once. If it is still unacceptable, repeat ICAL.
Calibration verification	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 85-115% for all analytes	Remake and reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until valid CV is obtained or ICAL is repeated. Samples analyzed since last acceptable CV must be reanalyzed.

CV = calibration verification
GC = gas chromatography
ICAL = initial calibration
ICV = initial calibration verification
RF = response factor
RSD = relative standard deviation
RT = retention time
%R = percent recovery

As previously stated, specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP and data validation checklist for each method.

SE 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all GC analyses. The QA Coordinators and Organics-GC Section Manager are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Organics-GC Section Manager is responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SE-4. Analytical QC samples are associated with field samples through the use of preparation batches. A preparation batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A preparation batch must not exceed 20 samples. Since most of the GC methods are multiple analyte methods, the acceptance criteria listed in Table SE-4 are general guidance values only because the specific criteria depend on the individual analyte and any special project requirements. More specific criteria are provided in the analytical method SOPs.

SE 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses and achieve precision and

accuracy equal to or better than the applicable reference method criteria (see DVC) for laboratory duplicates and laboratory control samples, respectively. In the absence of specific criteria, the general criteria listed in Table SE-4 are used.

SE 5.2 Analyte Identification

Target analytes must be identified by retention time (RT). RT windows for each target analyte peak must be centered on the RTs of the initial daily calibration verification. For a valid analyte identification, the RT for an analyte must be within the RT window. For second column confirmation, the analyte must be within the RT window on both columns.

SE 5.3 Blanks

A method blank (MB) is analyzed at the beginning of every analytical run and prior to the instrumental analysis of any samples. MB results are acceptable if the concentrations of all target analytes do not exceed the method detection limit (MDL) or reporting limit (RL).

If any target analyte concentration in the MB exceeds the MDL or RL, the source of contamination must be identified and eliminated. If contamination found in a MB is found in associated samples, the samples should be re-extracted. If it is not possible to re-extract the samples, the data must be appropriately qualified and the project manager notified. If contamination found in the method blank is not found in the associated samples, the samples' results may be reported, but the source of the contamination should still be investigated and eliminated.

Table SE-4. Summary of QC Requirements for GC Analysis

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration	Four (4) prepared samples analyzed prior to any sample analyses	Method criteria for LCS recovery and duplicate precision	Repeat until acceptable.
Retention times	For every analyte	Analyte peak within RT window	Compound identification is not valid if peaks are outside RT windows. If CV or LCS peaks are not in RT windows, system is out of control and must be corrected. Affected samples must be reanalyzed.
Method blank	Daily prior to sample analysis	Analytes < MDL or RL	Clean analytical system and repeat MB analysis. Identify and eliminate the source of contamination. Affected samples must be reanalyzed or the associated data flagged
Laboratory control sample	One (1) per preparation batch	Method criteria, Default: 70% $\leq$%R$\leq$130%	Investigate and identify the problem. If system is in control (e.g., MS acceptable and LCS result is isolated problem), no corrective action is needed. If system is out of control, repeat analysis of batch.
Matrix spike sample	One (1) per preparation batch	Method criteria, Default: 70% $\leq$% R$\leq$130%	Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch.
Surrogate spike	In every sample for applicable methods	Method criteria Default: 70% $\leq$% R$\leq$130%	Repeat instrumental analysis. If it is still unacceptable, investigate for possible matrix effect or extraction or system problem.
Internal standard	In every sample for applicable methods	50-200% %R compared to IS of preceding CV	Repeat instrumental analysis. If it is still unacceptable, investigate for possible matrix effect or extraction or system problem.
Sample duplicate or matrix spike duplicate	One (1) per preparation batch	Method criteria, Default: RPD $\leq$20% if analytes > RL	Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch.
Blind performance evaluation sample	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.

CV = calibration verification
DVC = data validation checklist
GC = gas chromatography
IS = internal standard
LCS = laboratory control sample
MB = method blank
PE = performance evaluation
QC = quality control
%R = percent recovery
RPD = relative percent difference
RT = retention time

SE 5.5 Laboratory Control Samples

A laboratory control samples (LCS) is second-source to the calibration standards and must be analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The LCS results are acceptable if the percent recovery of the target compounds are within the acceptance ranges listed in the data validation checklist or project specified limits.

If the LCS results do not meet specifications, the cause and impact of the problem must be determined. If the LCS results appear anomalous (e.g., bad injection, isolated extraction problem) and the matrix spike results and other batch QC are acceptable, the batch's samples' results are acceptable. If both the LCS and MS results are unacceptable, then instrumental (i.e., GC) analysis of the sample batch must be repeated. If after instrumental reanalysis both the LCS and MS are still unacceptable, then the sample batch must be re-extracted. If it is not possible to re-extract, the project manager must be notified. The project manager will contact the client and possibly arrange for recollection of samples.

SE 5.6 Matrix Spikes

Spikes are run every 10 to 20 samples depending on the specific method or per project requirements. Spike recoveries must fall within the acceptance ranges listed in the data validation checklist or project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause and impact of the problem must be determined. If the associated LCS results are acceptable, then the MS results are reportable with the qualifier that a matrix effect was observed. If the problem adversely affected the entire analysis batch, such as LCS results also unacceptable (see LCS discussion in SE5.5), all samples in the batch must be reanalyzed.

SE 5.7 Surrogate Spikes

For every sample analyzed, surrogate spike recoveries must also fall within acceptance ranges listed in the data validation checklist. If a sample's surrogate result is outside the acceptance range, the following procedures are necessary:

1) Check for calculation errors. If errors are found, recalculate the data.
2) Check chromatogram for interfering peaks.
3) Check instrument performance. If a problem is identified, correct the problem and reanalyze the sample.
4) If no instrument problem is found, the sample should be re-extracted and re-analyzed. If no sample remains, the client must be notified of the situation.
5) If the sample has been duplicated or spiked check for indication of problem due to matrix.

SE 5.8 Internal Standard

Internal standard (IS) recoveries must be between 50% and 200% compared to the IS of the preceding CV. If a sample's IS recovery is outside the acceptance range, follow the general corrective action procedures used for surrogate compounds.

SE 5.9 Duplicates

A duplicate sample or duplicate matrix spike sample is analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD) must be within the acceptance ranges listed in the data validation checklist or project specified limits.

If the QC criteria for duplicate sample or duplicate spike analyses are not satisfied, the cause and impact of the problem must be determined. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SE 5.10 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results are received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem.

SE 6.0 Data Management

SE 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to QC requirements specified for each type of analysis (Section SE5.0).

SE 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis. The analyst then enters the results for both samples and QC measurements into the laboratory information management system (LIMS).

Deviations from the specified data reduction procedures are permitted only with approval of the Organics-GC Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SE 6.3 Data Validation

As stated in QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and

qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary, and completed data validation checklist
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented with a signed data validation checklist. The checklist is archived in the associated data package/file.

SE 6.4 Data Reporting

After peer review of the data is completed as described in Section SE6.3 and the results are approved a preliminary report is generated. The applicable Project Manager (PM) reviews the preliminary report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections, then a final report is produced. The final report (QAM Exhibit 7-2) is also reviewed and signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Work Order No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports as illustrated in QAM Table 7-1. For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 3 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long-term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. CTL's Information System personnel maintain the LIMS on-site. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement F: Organic Analysis by Gas Chromatography/Mass Spectrometry

This supplement to the CTL's Quality Assurance Manual (QAM) describes the quality control (QC) requirements, procedures, and measurements utilized in performing organic analyses by gas chromatography/mass spectrometry (GC/MS). These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SF 1.0 Data Quality Objectives

The general laboratory DQOs are listed in Section 1.2 of the CTL QAM. How each of the general DQO categories are assessed for organic analyses by GC/MS and what are the nominal QC acceptance criteria for associated QC specifications are described in the remainder of this section. Tables SF-1 and SF-2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical methods and/or in the project plan.

SF 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see QAM Section 4.4).

SF 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing matrix spike (MS) samples, laboratory control samples (LCSs), and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SF-1 and SF-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurement's results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SF 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of MS samples, and replicate analyses of LCSs. Results from these measurements are compared to the criteria listed in Tables SF-1 and SF-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Organic Analysis by Gas Chromatography/ Mass Spectrometry

SF 1.0 Data Quality Objectives

SF 2.0 Method Requirements

SF 3.0 Instrument Testing, Inspection, and Maintenance Requirements Data Usability

SF 4.0 Instrument Calibration and Frequency

SF 5.0 Quality Control

SF 6.0 Data Management

Table SF-1. DQOs for Organic Analyses by Gas Chromatography/Mass Spectrometry of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/L)	Completeness (%)
Petroleum hydrocarbons	70-130	70-130	≤20	0.5	100
Semi-volatile organic compounds (SV)	70-130	70-130	≤20	10	100
Volatile organic compounds (VOCs)	70-130	70-130	≤20	0.5	100

LCS = laboratory control sample
MS = matrix spike
% R = percent recovery
MDL = method detection limit
% RSD = percent relative standard deviation
RPD = relative percent difference

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

Table SF-2. DQOs for Organic Analyses by Gas Chromatography/Mass Spectrometry of Solid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/kg)	Completeness (%)
Petroleum hydrocarbons	70-130	70-130	≤20	10	100
Semi-volatile organic compounds (SV)	70-130	70-130	≤20	200	100
Volatile organic compounds (VOCs)	70-130	70-130	≤20	10	100

LCS = laboratory control sample
MS = matrix spike
% R = percent recovery
MDL = method detection limit
% RSD = percent relative standard deviation
RPD = relative percent difference

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

SF 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SF-1 and SF-2 or the project-specific MDLs.

SF 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SF 1.7 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SF 2.0 Method Requirements

SF 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g., Accu Standards, Absolute, Ultra Scientific, Chem Services). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in QAM Section 3.2. Other material must meet the minimum requirements outlined in the approved methodology.

SF 2.2 Criteria for Instruments

Each gas chromatograph/mass spectrometer used for organic analyses must meet all the requirements for the analytical method (see QAM Section 3.2.1) and must be equipped with the appropriate separation columns and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Operation of instruments must always be in accordance with manufacturers' and methods' instructions, and instrument performance criteria specified in a method must be met before analysis of any samples.

SF 2.3 Criteria for Analysis

GC-MS based organic analyses performed by the CTL laboratory include sample preparation/extraction methods, clean-up methods, and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section.

SF 2.3.1 Purge and Trap Extraction

Purge and trap extraction is used for extracting volatile organic compounds (VOCs) from water and soil samples, linked to analyses of VOCs by GC and GC/MS. The extraction is identical regardless of the measurement procedure. Helium is "purged" through the sample, stripping the volatiles and carrying them to a sorbent trap downstream. The analytes are thermally desorbed and backflushed into a GC where the components are separated and analyzed using a variety of detectors. Highly contaminated soil samples may be extracted with methanol prior to analysis. Highly contaminated water samples are often diluted prior to analysis.

SF 2.3.2 Liquid/Liquid Extraction-Separatory Funnel

Liquid/liquid extraction with separatory funnel is used to extract semivolatile organic compounds from liquid samples. The sample is pH-adjusted, if necessary, then extracted with methylene chloride in a separatory funnel. Residual water is removed from the extract with

sodium sulfate, then the extract is concentrated by evaporation. For analysis of pesticides and PCBs, the extract is solvent exchanged into hexane.

SF 2.3.3 Pressurized Fluid Extraction

Pressurized Fluid Extraction (PFE) is a procedure for extracting water insoluble or water slightly soluble semi-volatile organic compounds from clays, sediments, sludges and waste solids. The method uses an elevated temperature and pressure to achieve analyte recoveries equivalent to those from soxhlet extraction, using less solvent and taking significantly less time than the soxhlet procedure.

SF 2.3.4 TCLP / TCLP ZHE Preparation for Organics

Toxicity characteristic leading procedure (TCLP) utilizes either a standard or zero-headspace extraction vessel (ZHE) for determining the mobility of organic contaminants present in all types of environmental matrices. The ZHE device allows for liquid/solid separation, extraction, and final extract filtration without opening the vessel, so loss of volatiles is precluded. TCLP leachates are prepared from soil and solid waste samples containing minimal or no filterable fluids. Sample leachates are generated in an extraction vessel, using various extraction fluids, at amounts equal to 20 times the mass of the solid being leached. Following the 18 hour extraction, the TCLP extract is filtered, stored, and submitted for organics analysis.

SF 2.3.5 Volatile Organic Components

A GC/MS method used to determine volatile organic compounds (VOCs) in a variety of aqueous, soil, and solid waste matrices. Analaytes are separated on a capillary column in the GC and detected with a mass spectrometer (MS). The MS provides qualitative identifications through reference spectra or fingerprints unique to each compound. Identification is futher enhanced by comparing elution retention times to calibration standards. Compounds are quantitated by relating the relative mass spectrometer response for a selected unique ion to a corresponding response ion from an assigned internal standard. The method is often used as a substitute for GC methods without MS detection, such as for BTEX by GC-PID.

SF 2.3.6 Semi-volatile Organic Components

A GC/MS method used to determine semi-volatile organic compounds (SV) in a variety of aqueous, soil, and solid waste matrices. Analaytes are separated on a capillary column in the GC and detected with a mass spectrometer (MS). The MS provides qualitative identifications through reference spectra or fingerprints unique to each compound. Identification is futher enhanced by comparing elution retention times to calibration standards. Compounds are quantitated by relating the relative mass spectrometer response for a selected unique ion to a corresponding response ion from an assigned internal standard.

SF 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The Organics-GC/MS Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand to perform necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SF 4.0 Instrument Calibration and Frequency

General calibration requirements for organic analyses by GC/MS are summarized in Table SF-3. Calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process as described in Table SF-3 and corrective actions if requirements are not satisfied are applicable to all GC/MS analytical methods.

As previously stated, specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP for each method.

SF 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all GC/MS analyses. The QA Coordinators and Organics-GC/MS Section Manager are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Organics-GC/MS Section Manager is responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SF-4. Analytical QC samples are associated with field samples through the use of preparation batches. A preparation batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A preparation batch must not exceed 20 samples. Since most of the GC/MS methods are multiple analyte methods, the acceptance criteria listed in Table SF-4 are general guidance values only because the specific criteria depend on the individual analyte, individual method, and any special project requirements. More specific criteria are provided in the analytical method.

Table SF-3. GC/MS Calibration Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Mass spectrometer tuning	First step of every ICAL and CV	Satisfy method criteria	Correct problem and repeat until acceptable.
Three-point or five-point initial calibration plus blank	Initially and as needed	CCCs and SPCCs must satisfy method criteria. Individual response factors for calibration standards must exhibit either ≤15% RSD or r≥0.99 for regression line.	Correct problem and repeat until acceptable.
Retention time windows	With every ICAL	ISs should be selected so RRTs for most target analytes are 0.80-1.20 compared to appropriate ISs.	If analyte peaks are not within RT windows, recalibrate.
Calibration verification	Every 12 hours prior to sample analysis	RFs for SPCCs must satisfy method criteria. RFs for CCCs must exhibit <20% difference or drift from ICAL. Compared to ICAL mid-point standard, ISs' RTs must be within ±30 seconds and ISs' areas must be – 50% to +100%.	Remake and reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until valid CV obtained or ICAL is repeated. Must reanalyze samples analyzed since last acceptable CV.

CCC = calibration check compound
CV = calibration verification
GC/MS = gas chromatography/mass spectrometry
ICAL = initial calibration
ICV = initial calibration verification
%R = percent recovery
RF = response factor
RTT = relative retention time
RSD = relative standard deviation
RT = retention time
SPCC = system performance check compound

Table SF-4. Summary of QC Requirements for GC/MS Analysis

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration	Four (4) prepared samples analyzed prior to any sample analyses	Method criteria for LCS recovery and duplicate precision	Repeat until acceptable.
Retention times	For ISs and every analyte in every sample	ISs RTs within ±30 seconds of last CV RTs for targets ±0.06 RRT units compared to last CV	Compound identification is not valid if peak is outside RT window. If CV or LCS peaks are not in RT windows, system is out of control and must be corrected. Affected samples must be reanalyzed.
Method blank	Daily prior to sample analysis	Analytes < RL	Clean analytical system and repeat MB analysis. Identify and eliminate the source of contamination.
Laboratory control sample	One (1) per preparation batch	Method criteria default: 70% ≤%R≤130%	Investigate and identify the problem. If system in control (e.g., MS acceptable and LCS result is isolated problem), no corrective action is needed. If system is out of control, repeat analysis of batch.
Matrix spike sample	One (1) per preparation batch	Method criteria default: 70% ≤% R≤130%	Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch.
Surrogate spike	In every sample for applicable methods	Method criteria default: 70% ≤% R≤130%	Repeat instrumental analysis. If it is still out, investigate for possible matrix effect or extraction or system problem.
Internal standard	In every sample for applicable methods	50-200% %R compared to IS of preceding CV	Repeat instrumental analysis. If it is still out, investigate for possible matrix effect or extraction or system problem
Sample duplicate or matrix spike duplicate	One (1) per preparation batch	Method criteria default: RPD ≤20% if analytes > RL	Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch.
Blind performance evaluation sample	Samples and frequency determined by accrediting agencies and projects	Determined by PE provider	Investigate all unacceptable results.

CV = calibration verification
%R = percent recovery
GC/MS = gas chromatography/mass spectrometry
IS = internal standard
LCS = laboratory control sample
MB = method blank
PE = performance evaluation
QC = quality control
RL = reporting limit
RPD = relative percent difference
RRT = relative retention time
RT = retention time

SF 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses and achieve precision and accuracy equal to or better than the applicable reference method criteria for laboratory duplicates and laboratory control samples, respectively. In the absence of specific criteria, the general criteria listed in Table SF-4 are used.

SF 5.2 Analyte Identification

Target analytes must be identified by retention time (RT) and mass spectrum. The intensities of characteristic ions of an analyte in a sample's mass spectrum must maximize in the same scan or within one scan of each other. The relative intensities of characteristic ions for the analyte in a sample's mass spectrum must agree with 30% of the relative intensities of those ions in the reference spectrum.

SF 5.3 Blanks

A method blank (MB) is analyzed at the beginning of every analytical run and prior to the instrumental analysis of any samples. MB results are acceptable if the concentrations of all target analytes do not exceed the reporting limit (RL).

If any target analyte concentration in the MB exceeds the RL, the source of contamination must be identified and eliminated. If contamination found in a MB is found in associated samples, the samples should be re-extracted. If it is not possible to re-extract the samples, the data must be appropriately qualified and the project manager notified. If contamination found in the method blank is not found in the associated samples, the samples' results may be reported, but the source of the contamination should still be investigated and eliminated.

SF 5.4 Laboratory Control Samples

A laboratory control samples (LCS) is second-source to the calibration standards and must be analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The LCS results are acceptable if the percent recovery of the target compounds are within the acceptance ranges or project specified limits.

If the LCS results do not meet specifications, the cause and impact of the problem must be determined. If the LCS results appear anomalous (e.g., bad injection, isolated extraction problem) and the matrix spike results and other batch QC are acceptable, the batch's samples' results are acceptable. If both the LCS and MS results are unacceptable, then instrumental (i.e., GC/MS) analysis of the sample batch must be repeated. If after instrumental reanalysis both the LCS and MS are still unacceptable, then the sample batch must be re-extracted. If it is not possible to re-extract, the project manager must be notified. The project manager will contact the client and possibly arrange for recollection of samples.

SF 5.5 Matrix Spikes

Spikes are run every 10 to 20 samples depending on the specific method or per project requirements. Spike recoveries must fall within the acceptance ranges or project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause and impact of the problem must be determined. If the associated LCS results are acceptable, then the MS results are reportable with the qualifier that a matrix effect was observed. If the problem adversely affected the entire analysis batch,

such as LCS results also unacceptable (see LCS discussion in SF5.4), all samples in the batch must be reanalyzed.

SF 5.6 Surrogate Spikes

For every sample analyzed, surrogate spike recoveries must also fall within acceptance. If a sample's surrogate result is outside the acceptance range, the following procedures are necessary:

1) Check for calculation errors. If errors are found, recalculate the data.
2) Check chromatogram for interfering peaks.
3) Check instrument performance. If a problem is identified, correct the problem and reanalyze the sample.
4) If no instrument problem is found, the sample should be re-extracted and re-analyzed. If no sample remains, the client must be notified of the situation.
5) If the sample has been duplicated or spiked check for indication of problem due to matrix.

SF 5.7 Internal Standard

Internal standard (IS) recoveries must be between 50% and 200% compared to the IS of the preceding CV.

If a sample's IS recovery is outside the acceptance range, follow the general corrective action procedures used for surrogate compounds.

SF 5.8 Duplicates

A duplicate sample or duplicate matrix spike sample is analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD) must be within the acceptance ranges or project specified limits.

If the QC criteria for duplicate sample or duplicate spike analyses are not satisfied, the cause and impact of the problem must be determined. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SF 5.9 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results are received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem.

SF 6.0 Data Management

SF 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see Section SF5.0).

SF 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis

Deviations from the specified data reduction procedures are permitted only with approval of the Organics-GC/MS Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SF 6.3 Data Validation

As stated in QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented and archived in the associated data package/file.

SF 6.4 Data Reporting

After peer review of the data is completed as described in Section SF6.3 and the results are approved, the analyst approves the result in LIMS and a preliminary report is generated. The applicable Project Manager (PM) reviews the preliminary report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections, then a final report is produced. The final report (QAM Exhibit 7-2) is also reviewed and signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Work Order No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports as illustrated in QAM Table 7-1. For the levels II, III, and IV deliverables formats the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 3 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long-term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. CTL's Information System personnel maintain the LIMS on-site. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement G: Analysis of Air Contaminant Sampling Media

This supplement describes the quality control (QC) requirements, procedures, and measurements utilized in performing analysis of air sampling sorbent tubes, and surface wipes. These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SG 1.0 Data Quality Objectives

The general laboratory DQOs are listed in CTL QAM Section 1.2. How each of the general DQO categories are assessed for air contaminant media analysis, and what are the nominal QC acceptance criteria for associated QC specifications, are described in the remainder of this section. Tables SG1 summarizes the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical method and/or in the project plan.

SG 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, surfaces) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see CTL QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see CTL QAM Section 4.4).

SG 1.2 Accuracy

Accuracy as percent recovery (%R) must be assessed by analyzing LCSs and single-blind or double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Table SG1 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SG 1.3 Precision

Precision is assessed by analyzing field duplicates (when available) and replicate analyses of laboratory control samples (LCS). Results from these measurements are compared to the criteria listed in Table SG1 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Analysis of Air Contaminant Sampling Media

SG 1.0 Data Quality Objectives

SG 2.0 Method Requirements

SG 3.0 Instrument Testing, Inspection and Maintenance Requirements

SG 4.0 Instrument Calibration and Frequency

SG 5.0 Quality Control

SG 6.0 Data Management

Table SG1. DQOs for Analysis of Air Contaminants on Prescribed Media

Medium and Analyte(s)	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	RL	Completeness (%)
Sorbent Tubes:					
Benzene	70 - 130	70 - 130	≤ 25	1 ppm[e]	100
Ethylbenzene	70 - 130	70 - 130	≤ 25	1 ppm[e]	100
Toluene	70 - 130	70 - 130	≤ 25	1 ppm[e]	100
Xylene (Total)	70 - 130	70 - 130	≤ 25	1 ppm[e]	100
Total VOCs (as product)	70 - 130	70 - 130	≤ 25	1 ppm[e]	100
Surface Wipes:					
Lead	90-110	80-120	≤ 20	2.5 ug/wipe[f]	100

a *Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.*
b. *Typical values listed. Exact values depend on specific method and compound.*
c. *Estimated RL in ug per medium; reported RLs are based on actual air volume sampled..*
d. *RL estimated assuming 8 hr. Sampling period, desorption volume of 2 ml, and achieving 90% desorption efficiency.*
e. *RL estimated assuming 50/100mg tube desorbed with 1 ml eluent, achieving 95% desorption efficiency.*
f. *Estimated RL in ug per wipe; reported RLs are based on actual surface area sampled..*

LCS = laboratory control sample
% R = percent recovery
MS = matrix spike
% RSD = percent relative standard deviation
RPD = relative percent difference
MDL = method detection limit
RL = reporting limit

SG 1.4 Quantitation/Reporting Limits

Quantitation limits for air or surface monitoring are dependent on several factors, including air volume or surface area sampled, sorbent tube size, desorption eluent volume, and desorption efficiency. Desorption efficiency is dependent on the compound of interest, and may vary with charcoal lots. Estimates of reporting limit (RL) capability are listed in Table SG1. RLs in analytical reports are calculated to reflect all pertinent sampling information.

SG 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SG 1.6 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SG 2.0 Method Requirements

SG 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g., Solutions Plus, ChemService, Fisher Scientific). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials where possible. Secondary standards for calibration and QC measurements are prepared from primary standards or an alternative certified source. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in CTL QAM Section 3.2. Other material used must meet the minimum requirements outlined in the approved methodology.

SG 2.2 Criteria for Instruments

The laboratory instruments utilized for air analysis must meet all the requirements for the analytical methods to be conducted (see CTL QAM Section 3.2.1) and must be equipped with the appropriate nebulizers, columns, detectors, as required. The specific equipment components and set-up requirements are described in the CTL SOP for each analytical method. Instruments operation must always be in accordance with manufacturers' and methods' instructions, and performance criteria specified in the methods must be met before analysis of any samples.

SG 2.3 Criteria for Analysis

Air contaminant analyses performed by the CTL laboratory include sample preparation/extraction and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section.

SG 2.3.1 Sorbent Tubes

Sorbent tubes, routinely used for sampling gasses and vapors from air in designated areas, are analyzed by NIOSH methods. Samples collected by pulling air through the tube packed with sorbent media (e.g., charcoal) are desorbed with an appropriate solvent prior to analysis.

SG 2.3.2 Surface Wipes

The entire wipe is placed in a digestion vessel to be prepared by hot acid digestion using nitric and hydrochloric acids. The acid digestion solublizes trapped metals and stabilizes them in their oxidized form. The digestate is diluted before being analyzed by inductively-coupled plasma atomic emission spectroscopy. After correction for background and interferences, the net emission signals are compared to the net emission of known standards to determine a concentration value for each metal of interest. The calculated concentration can then be related back to sample collection data to provide a concentration based on the area of surface wiped.

SG 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The applicable Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs (e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SG 4.0 Instrument Calibration and Frequency

Specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method.

General calibration requirements of Metals analyses are summarized in Tables SG2a. General calibration requirements of Organics analyses are summarized in Table SG2b. As previously stated, calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Tables SG2a b, and corrective actions if requirements are not satisfied, are applicable to all analytical methods, as appropriate.

Table SG2a. General Metals Instrument Calibration Requirements: ICP

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Linear dynamic range study	Annually	r≥0.995 for regression line	Repeat until acceptable
Two-point initial calibration plus blank	Initially and as needed	r≥0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R 90-110% for all analytes	Reanalyze ICV standard once. If it is still unacceptable, repeat ICAL.
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	< MDL or RL	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Calibration verification (CV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 90-110% for all analytes	Reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until a valid CV is obtained or ICAL is repeated. Must reanalyze samples analyzed since last acceptable CV.
Calibration Blank (CB)	Daily, prior to sample analysis, after every 10 samples, and at end of run	< MDL or RL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or ICAL repeated.

%R = percent recovery
RSD = relative standard deviation
MDL or RL= method detection limit or reporting limit

Table SG2b. General Organics Instrument Calibration Requirements: GC

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Five-point initial calibration plus blank (ICAL)	Initially and as needed	≤ 20% RSD for individual response factors or r≥0.99 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R 70-130% for all analytes	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After each ICAL, prior to sample analysis	< RL	Remake and reaanalyze ICV standard once, if still unacceptable repeat ICAL
Calibration verification (CV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 70-130% for all analytes	Remake and reanalyze CV once, if still unacceptable investigate and correct problem, cannot proceed until valid CV obtained or ICAL repeated
Calibration Blank (CB)	Daily, prior to sample analysis	< RL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem, cannot proceed until valid CB obtained or ICAL repeated

%R = percent recovery
RSD = relative standard deviation

SG 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all applicable analyses. The QA Coordinators and Analytical Section Managers are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, and duplicates. The Analytical Section Managers are responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Tables SG3a-b. Analytical QC samples are associated with field samples through the use of analysis batches. An analysis batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. An analysis batch must not exceed 20 field samples. More specific criteria are provided in the individual analytical method.

Table SG3a. Summary of QC Requirements for Air Monitoring: Metals

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration sample (IDC)	Four (4) prepared standards that are analyzed one time prior to any sample analyses	In-house or method criteria for IDC recovery and precision	Repeat until acceptable
Method blank (MB)	Daily, using Corresponding sampling media, prior to sample analysis	Analytes < RL	Clean analytical system, repeat until MBs are in control
Sample duplicate (DUP), when available	One (1) per analytical batch	In-house derived limits Default: RPD $\leq$20% if analytes > RL	Investigate problem; if system precision out of control qualify results
Laboratory control sample (LCS)	Three (3) per analytical batch	90% $\leq$%R$\leq$110%	Halt analysis, fix problem, repeat associated sample analyses
Blind laboratory control sample (BLCS)	Three (3) per analytical batch	90% $\leq$%R$\leq$110%	Halt analysis, fix problem, repeat associated sample analyses
%R = percent recovery RPD = relative percent difference RL = reporting limit			

Table SG3b. Summary of QC Requirements for Air Monitoring: Organics

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Capability (IDC)	Four (4) prepared standards (in CS_2) that are analyzed one time prior to any sample analyses	In-house determined criteria for LCS recovery and duplicate precision	Repeat until acceptable
Method blank (MB)	Daily, using Corresponding sampling media, prior to sample analysis	Analytes < RL	Clean analytical system, repeat until MBs are in control
Sample duplicate (DUP), when available	One (1) per analytical batch	In-house derived limits Default: RPD $\leq$20% if analytes > RL	Investigate problem; if system precision out of control qualify results
Laboratory control sample (LCS)	One (1) per analytical batch	70% $\leq$%R$\leq$130%	Halt analysis, fix problem, repeat associated sample analyses
Blind laboratory control sample (BLCS)	Three (3) per analytical batch	90% $\leq$%R$\leq$110%	Halt analysis, fix problem, repeat associated sample analyses
%R = percent recovery RPD = relative percent difference RL = reporting limit			

SG 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be acceptably demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses of a known standard and achieve precision and accuracy equal to or better than the most recent in-house determined acceptance ranges for laboratory duplicates and laboratory control samples, respectively. This demonstration should be done in the appropriate matrix where possible. In the absence of in-house determined criteria, the general criteria listed in Tables SG3a-b are used.

SG 5.2 Blanks

A method blank (MB) is prepared in the appropriate matrix at a frequency of one per 20 field samples depending on the specific method or project requirements. The MB is analyzed at the beginning of every instrumental analytical run and prior to the analysis of any samples. MB results are acceptable if the concentrations of the target analyte does not exceed the reporting limit (RL). If any target analyte concentration in the MB exceeds the RL, the source of contamination must be identified and eliminated. Analysis of samples cannot proceed until a compliant MB is obtained.

SG 5.3 Duplicates

A duplicate sample (DUP) can only be analyzed for an air or surface wipe sample if duplicate sampling has been conducted in the field. In the event that duplicate collections are available, duplicates are analyzed. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, must be within the in-house determined acceptance ranges listed in the CTL SOPs, or project specified limits.

If the QC criteria for duplicate sample analyses are not satisfied, the cause of the problem must be investigated. All data associated must be appropriately qualified in the data file and in the report. Sample recollection is at the discretion of the client.

SG 5.4 Laboratory Control Samples

A laboratory control sample (LCS) is second-source to the calibration standards and must be analyzed at a frequency of three per every 20 field samples depending on the specific method or project requirements. The LCS results are acceptable if the percent recovery of each analyte is within the in-house determined acceptance range or project specified limits. If the LCS results do not meet specification, the cause of the problem must be investigated, and all sample analyses in the associated batch must be appropriatedly qualified.

SG 5.5 Blind Laboratory Control Samples

A blind laboratory control sample (BLCS) is second-source to the calibration standards and must be analyzed at a frequency of three per every 20 field samples, depending on the specific method or project requirements. BLCS are blind (to the analysis) conducting the testing. The BLCS results are acceptable if the percent recovery of each analyte is within the in-house determined acceptance range or project specified limits. If the LCS results do not meet specification, the cause of the problem must be investigated, and all sample analyses in the associated batch must be appropriatedly qualified

SG 6.0 Data Management

SG 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to the QC requirements specified for each type of analysis (see SG 5.0).

SG 6.2 Data Reduction

Detailed procedures for converting raw data to final reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis.
Deviations from the specified data reduction procedures are permitted only with approval of the applicable Analytical Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SG 6.3 Data Validation

As stated in CTL QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data, calculations or calculation records, calibration data or records, QC measurement results, test results summary
- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented and archived in the associated data package/file.

SG 6.4 Data Reporting

After peer review of the data is completed as described in Section SG 6.3 and the results are approved, the analyst approves the result in LIMS and a report is generated. The applicable Project Manager (PM) reviews the report (CTL QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections. The final report (CTL QAM Exhibit 7-2) is signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Lab No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports (illustrated in QAM Table 7-1). For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 3 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. The LIMS is maintained on-site by CTL's Information System personnel. Full server backups are performed nightly. Other electronic data include instrument magnetic

tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

Supplement H: Organic Analysis by High Performance Liquid Chromatography

This supplement to the CTL's Quality Assurance Manual (QAM) describes the quality control (QC) requirements, procedures, and measurements utilized in performing organic analyses by high performance liquid chromatography (HPLC). These QC activities are designed to assess and monitor the quality of analytical data generated and the ability of that data to satisfy QC acceptance criteria and data quality objectives (DQOs).

SH 1.0 Data Quality Objectives

The general laboratory DQOs are listed in Section 1.2 of the CTL QAM. How each of the general DQO categories are assessed for organic analyses by HPLC and what are the nominal QC acceptance criteria for associated QC specifications are described in the remainder of this section. Tables SH-1 and SH-2 summarize the nominal QC acceptance criteria for the types of analyses performed. It should be noted that the listed QC acceptance criteria are nominal (i.e., default or interim) and may not apply to all individual compounds or projects. When the nominal criteria do not apply, the specific criteria are listed in the analytical and/or in the project plan.

SH 1.1 Representativeness

Measurements on a sample are made so the final results are as representative as possible of the media (e.g., air, soil, water) sampled and conditions being measured. Representativeness of the measurement in the laboratory is attained by conducting the analysis by the appropriate analysis method (see QAM Section 3.2.1) and completing the analysis within the applicable analysis holding time (see QAM Section 4.4).

SH 1.2 Accuracy

Accuracy as percent recovery (%R) is assessed by analyzing matrix spike (MS) samples, laboratory control samples (LCSs), and single-blind and double-blind performance evaluation (PE) samples. Results from these measurements are compared to the criteria listed in Tables SH-1 and SH-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

SH 1.3 Precision

Precision is assessed by analyzing laboratory duplicates, replicate analyses of MS samples, and replicate analyses of LCSs. Results from these measurements are compared to the criteria listed in Tables SH-1 and SH-2 or, if more specific criteria apply, to the criteria in the applicable method. These QC measurements' results are used to demonstrate acceptable method performance or to trigger corrective action when criteria are not satisfied.

Table SH-1. DQOs for Organic Analyses by HPLC of Liquid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/L)	Completeness (%)
Polynuclear aromatic hydrocarbons (PAHs)	70-130	70-130	≤ 20	0.01-1	100
Nitro-explosives	70-130	70-130	≤ 20	0.01-20	100

LCS = laboratory control sample
MS = matrix spike
% R = percent recovery
MDL = method detection limit
% RSD = percent relative standard deviation
RPD = relative percent difference

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

Table SH-2. DQOs for Organic Analyses by HPLC of Solid Samples

Type of Analysis	Accuracy[a] (LCS %R)	Accuracy[a] (MS %R)	Precision[a] (% RSD or RPD)	MDL[b] (µg/kg)	Completeness (%)
Polynuclear aromatic hydrocarbons (PAHs)	70-130	70-130	≤ 20	0.5-50	100
Nitro-explosives	70-130	70-130	≤ 20	0.3-3	100

LCS = laboratory control sample
MS = matrix spike
% R = percent recovery
MDL = method detection limit
% RSD = percent relative standard deviation
RPD = relative percent difference

a. Criteria listed are default values. Exact values depend on specific method, compound, and project. Criteria apply to concentrations ≥ RL.
b. Typical values listed. Exact values depend on specific method and compound and must be ≤ RL.
c. Typical values listed. Exact values depend on specific method, compound and project.

SH 1.4 Detectability

Method detection limits are determined or verified at least annually using the procedure and calculation described in QAM Section 7.2.4. The MDLs must be less than or equal to the values listed in Tables SH-1 and SH-2 or the project-specific MDLs.

SH 1.5 Completeness

Laboratory analysis completeness is expressed as the number of samples analyzed with valid results (i.e., data that meet all specifications) as a percent of the total number of samples submitted for analyses. One hundred percent of the total number of samples submitted for analysis must result in valid analytical data or the customer must be notified and consulted to determine the corrective action (e.g., recollection of sample).

SH 1.6 Comparability

The comparability of CTL data sets to those generated by different laboratories shall be achieved through use of standardized methods, traceable standards, and by participation in PEs.

SH 2.0 Method Requirements

SH 2.1 Criteria for Standards and Material

Primary standards are purchased from the best available source (e.g., Accu Standards, Absolute, Ultra Scientific, Chem Services). All commercially manufactured standards must be certified by the manufacturer to be traceable to National Institute of Standards and Technology (NIST) reference materials. Certified standards from at least two independent manufacturers must be on hand at all times. Secondary standards for calibration and QC measurements are prepared from primary standards. Detailed instructions for preparation of secondary standards are provided in the applicable analytical method SOPs. Traceability of the secondary standards is maintained by ensuring that equipment used for secondary standard preparation is maintained, operated and calibrated according to the requirements in QAM Section 3.2. Other material used must meet the minimum requirements outlined in the approved methodology.

SH 2.2 Criteria for Instruments

Each liquid chromatograph used for organic analyses must meet all the requirements for the analytical method (see QAM Section 3.2.1) and must be equipped with the appropriate separation columns and detectors. The specific equipment components and set up requirements are described in the CTL SOP for each analytical method. Operation of instruments must always be in accordance with manufacturers' and methods' instructions, and instrument performance criteria specified in a method must be met before analysis of any samples.

SH 2.3 Criteria for Analysis

HPLC-based organic analyses performed by the CTL laboratory include sample preparation/extraction methods, clean-up methods, and instrumental analysis methods. Detailed instructions for analysis of samples and reduction of data are provided in the applicable CTL SOPs. Brief descriptions of the methods utilized are listed in the remainder of this section.

SH 2.3.1 Liquid/Liquid Extraction-Separatory Funnel

Liquid/liquid extraction with separatory funnel is used to extract semivolatile organic compounds from liquid samples. The sample is pH-adjusted, if necessary, then extracted with methylene chloride in a separatory funnel. Residual water is removed from the extract with sodium sulfate, then the extract is concentrated by evaporation.

SH 2.3.2 Pressurized Fluid Extraction

Pressurized Fluid Extraction (PFE) is a procedure for extracting water insoluble or water slightly soluble semi-volatile organic compounds from clays, sediments, sludges and waste solids. The method uses an elevated temperature and pressure to achieve analyte recoveries equivalent to those from soxhlet extraction, using less solvent and taking significantly less time than the soxhlet procedure.

SH 2.3.3 Solid-Phase Extraction

Solid-phase extraction (SPE) is used to extract semivolatile and other extractable organic compounds from aqueous samples. A measured volume of sample is adjusted to a specified pH and then extracted using a SPE device. Target analytes are eluted from the solid-phase media using an appropriate solvent. The concentrated extract may be exchanged into a solvent compatible with determinative procedures employed for the measurement of the target analytes.

SH 2.3.4 Ultrasonic Extraction

Ultrasonic extraction is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids, such as soils, sludges and solid wastes. A measured volume of sample is mixed with a drying agent to form a free flowing powder. This is solvent extracted three times using ultrasonic extraction. The resulting extract is ready for clean-up and/or analysis following concentration.SH 2.3.4

SH 2.3.5 Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC

This is a HPLC method to determine selected PAHs based compounds in a variety of matrices. This method is applicable to nearly all types of samples including, but not limited to, ground water, waste solvents, soils, and sediments. solvent extracts are applied onto a column in a HPLC equipped with UV and fluorescence detectors. Qualitative identifications are attained by analyzing reference standards under the same conditions used for samples and by comparing resultant HPLC retention times.

For all USACOE proposals and quotations, the following statement must be provided to the Client:

US-ACOE Required Disclaimer for SW-846 Method 8310: *Analysis of PAHs by current EPA Method 8310 does not provide for the confirmation of analytes, and therefore may result in the detection of false positives.*

SH 2.3.6 Nitro-explosives by HPLC

This is a HPLC method to determine nitro based explosives (Nirtoaromatics, Nitramines and Nitroglycerine) in water and soil.. Extracts are applied onto a column in a HPLC equipped with UV detector. Qualitative identifications are attained by analyzing reference standards under the same conditions used for samples and by comparing resultant HPLC retention times.

SH 3.0 Instrument Testing, Inspection, and Maintenance Requirements

All maintenance is conducted in accordance with manufacturers' recommendations. These requirements are described in CTL standard operating procedures and appropriate instrument maintenance manuals. The Analytical Section Manager is responsible for ensuring that timely maintenance is conducted and that sufficient spare parts are on hand for necessary maintenance and repair procedures.

The frequency of maintenance performed depends on the equipment; laboratory maintenance is scheduled and conducted daily, monthly, weekly, quarterly, semiannually, and annually, as needed. A few maintenance needs

(e.g., accidental breakage, part failure) are not covered by the general maintenance schedule, and such maintenance is performed as needed.

SH 4.0 Instrument Calibration and Frequency

General calibration requirements for organic analyses by HPLC are summarized in Table SH-3. Calibration requirements for individual methods may differ slightly from the listed general requirements, but the components of the calibration process described in Table SH-3 and corrective actions if requirements are not satisfied are applicable to all HPLC analytical methods.

Table SH-3. HPLC Calibration Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Three-point or five-point initial calibration plus blank	Initially and as needed	Individual response factors for standards must exhibit either $\leq$20% RSD or r$\geq$0.99 for regression line.	Correct problem and repeat until acceptable.
Retention time windows	Initially and with every ICV	RT windows must be centered on ICV, established from three injections or other documented approach.	If analyte peaks are not within RT windows, reestablish RT windows or recalibrate.
Initial calibration verification	After each ICAL, prior to sample analysis	%R 85-115% for all analytes	Remake and reanalyze ICV standard once. If it is still unacceptable, repeat ICAL.
Calibration verification	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R 85-115% for all analytes	Remake and reanalyze CV once. If it is still unacceptable, investigate and correct problem. Analysis cannot proceed until valid CV is obtained or ICAL is repeated. Samples analyzed since last acceptable CV must be reanalyzed.

CV = calibration verification
HPLC = gas chromatography
ICAL = initial calibration
ICV = initial calibration verification
RF = response factor
RSD = relative standard deviation
RT = retention time
%R = percent recovery

As previously stated, specific instrument calibration requirements can and do vary slightly depending on the particular method and the project and regulatory requirements for the project. Detailed descriptions of specific calibration requirements are provided in the CTL analytical method SOP.

SH 5.0 Quality Control

To ensure that data of known and documented quality are generated, the QC criteria described in this section must be met for all HPLC analyses. The QA Coordinators and Section Manager are responsible for monitoring and documenting procedure performance, including the analysis of control samples, blanks, matrix spikes, and duplicates. The Section Manager is responsible for implementing corrective actions when acceptable procedure performance, as described in this section, is not met.

Specific QC samples and frequencies are summarized in Table SH-4. Analytical QC samples are associated with field samples through the use of preparation batches. A preparation batch is defined as a suite of samples of a similar matrix that are processed as a unit within a specific time period. A preparation batch must not exceed 20 samples. Since most of the HPLC methods are multiple analyte methods, the acceptance criteria listed in Table

SH-4 are general guidance values only because the specific criteria depend on the individual analyte and any special project requirements. More specific criteria are provided in the analytical method SOPs.

SH 5.1 Demonstration of Capability

The capability to acceptably perform an analytical method must be demonstrated by each analyst prior to conducting sample analyses. The analyst must conduct four replicate analyses and achieve precision and accuracy equal to or better than the applicable reference method criteria for laboratory duplicates and laboratory control samples, respectively. In the absence of specific criteria, the general criteria listed in Table SH-4 are used.

SH 5.2 Analyte Identification

Target analytes must be identified by retention time (RT). RT windows for each target analyte peak must be centered on the RTs of the initial daily calibration verification. For a valid analyte identification, the RT for an analyte must be within the RT window. For second column confirmation, the analyte must be within the RT window on both columns.

SH 5.3 Blanks

A method blank (MB) is analyzed at the beginning of every analytical run and prior to the instrumental analysis of any samples. MB results are acceptable if the concentrations of all target analytes do not exceed the reporting limit (RL).

If any target analyte concentration in the MB exceeds the RL, the source of contamination must be identified and eliminated. If contamination found in a MB is found in associated samples, the samples should be re-extracted. If it is not possible to re-extract the samples, the data must be appropriately qualified and the project manager notified. If contamination found in the method blank is not found in the associated samples, the samples' results may be reported, but the source of the contamination should still be investigated and eliminated.

Table SH-4. Summary of QC Requirements for HPLC Analysis

QC Sample or Activity	Minimum Frequency	Acceptance Criteria	Corrective Action
Capability demonstration	Four (4) prepared samples analyzed prior to any sample analyses	Method criteria for LCS recovery and duplicate precision	Repeat until acceptable.
Retention times	For every analyte	Analyte peak within RT window	Compound identification is not valid if peaks are outside RT windows. If CV or LCS peaks are not in RT windows, system is out of control and must be corrected. Affected samples must be reanalyzed.
Method blank	Daily prior to sample analysis	Analytes < RL	Clean analytical system and repeat MB analysis. Identify and eliminate the source of contamination.
Laboratory control sample	One (1) per preparation batch	Method criteria default: 70% $\leq$%R$\leq$130%	Investigate and identify the problem. If system is in control (e.g., MS acceptable and LCS result is isolated problem), no corrective action is needed. If system is out of control, repeat analysis of batch.
Matrix spike sample	One (1) per preparation batch	Method criteria default: 70% $\leq$% R$\leq$130%	Investigate problem. If system accuracy is in control, qualify results. If system accuracy is out of control, reanalyze entire batch.
Surrogate spike	In every sample for applicable methods	Method criteria default: 70% $\leq$% R$\leq$130%	Repeat instrumental analysis. If it is still unacceptable, investigate for possible matrix effect or extraction or system problem.
Sample duplicate or matrix spike duplicate	One (1) per preparation batch	Method criteria default: RPD $\leq$20% if analytes > RL	Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch.

CV = calibration verification
HPLC = high performance liquid chromatography
QC = quality control
IS = internal standard
LCS = laboratory control sample
MB = method blank
%R = percent recovery
RL = reporting limit
RPD = relative percent difference
RT = retention time

SH 5.5 Laboratory Control Samples

A laboratory control samples (LCS) is second-source to the calibration standards and must be analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The LCS results are acceptable if the percent recovery of the target compounds are within the acceptance ranges or project specified limits.

If the LCS results do not meet specifications, the cause and impact of the problem must be determined. If the LCS results appear anomalous (e.g., bad injection, isolated extraction problem) and the matrix spike results and other batch QC are acceptable, the batch's samples' results are acceptable. If both the LCS and MS results are unacceptable, then instrumental (i.e., HPLC) analysis of the sample batch must be repeated. If after instrumental reanalysis both the LCS and MS are still unacceptable, then the sample batch must be re-extracted. If it is not possible to re-extract, the project manager must be notified. The project manager will contact the client and possibly arrange for recollection of samples.

SH 5.6 Matrix Spikes

Spikes are run every 10 to 20 samples depending on the specific method or per project requirements. Spike recoveries must fall within the acceptance ranges or project specified limits.

If the QC criteria for the matrix spike analyses are not satisfied, the cause and impact of the problem must be determined. If the associated LCS results are acceptable, then the MS results are reportable with the qualifier that a matrix effect was observed. If the problem adversely affected the entire analysis batch, such as LCS results also unacceptable (see LCS discussion in SH 5.5), all samples in the batch must be reanalyzed.

SH 5.7 Surrogate Spikes

For every sample analyzed, surrogate spike recoveries must also fall within acceptance ranges. If a sample's surrogate result is outside the acceptance range, the following procedures are necessary:

1) Check for calculation errors. If errors are found, recalculate the data.
2) Check chromatogram for interfering peaks.
3) Check instrument performance. If a problem is identified, correct the problem and reanalyze the sample.
4) If no instrument problem is found, the sample should be re-extracted and re-analyzed. If no sample remains, the client must be notified of the situation.
5) If the sample has been duplicated or spiked check for indication of problem due to matrix.

SH 5.8 Duplicates

A duplicate sample or duplicate matrix spike sample is analyzed at a frequency of at least one per 20 samples depending on the specific reference method or project requirements. The relative percent difference (RPD) between duplicate samples, for samples having analyte concentrations greater than their respective reporting limit, or between a matrix spike (MS) and matrix spike duplicate (MSD) must be within the acceptance ranges or project specified limits.

If the QC criteria for duplicate sample or duplicate spike analyses are not satisfied, the cause and impact of the problem must be determined. If the problem adversely affected the entire analysis batch, all samples in the batch must be reanalyzed.

SH 5.10 Blind Performance Evaluation Samples

CTL participates in numerous performance evaluation (PE) studies to obtain an independent assessment of the accuracy of its analyses, to fulfill specific project requirements, and to maintain laboratory accreditations. All PE analyses performed by CTL are performed by the same analysts and using the same procedures that are used for routine sample analyses for the analyte(s) of interest. The PE results must satisfy the PE acceptance criteria specified by the PE provider or project. After an evaluation of the PE results are received, any results outside of acceptance limits are investigated and corrective actions taken to prevent recurrence of the problem.

SH 6.0 Data Management

SH 6.1 Data Generation

Sample analyses at the CTL laboratory are performed by qualified analysts and by using appropriate analytical methods to ensure the generated data are valid and legally defensible. Each laboratory analyst must successfully complete a prescribed sequence of training objectives before that individual is designated qualified and permitted to independently conduct any assignment or analyses (see QAM Section 2.3), and all analyses require strict adherence to QC requirements specified for each type of analysis (Section SH 5.0).

SH 6.2 Data Reduction

Detailed procedures for converting raw data to final, reportable results are provided in the analytical method SOPs. A hard copy printout of all raw data is reviewed, evaluated, edited if necessary, signed, and dated by the analyst who performed the analysis.

Deviations from the specified data reduction procedures are permitted only with approval of the Organics-HPLC Section Manager, and such deviations are to be recorded on the raw data recording forms. Normally a written description of the modification and the reason for it will be included in the final report.

SH 6.3 Data Validation

As stated in QAM Section 7.1.2, one hundred percent of the data must receive independent technical review. The reviewer must be a qualified individual other than the data generator (e.g., peer analyst or Analytical Section Manager). The independent technical reviewer must meet the minimum training and qualifications requirements for analysts. Individuals not qualified to perform data interpretation cannot perform independent technical review. The reviewer(s) must ensure that:

- Data generation and reduction were conducted in a technically correct manner in accordance with the methods used
- Data are reported in the proper units and with the correct number of significant figures
- Calculations were verified by a valid calculation program, a spot check of verified calculation programs, or 100% check of all hand calculations
- All variances from an accepted method and the rationale for the variations were documented and approved
- Data were reviewed for transcription errors
- Analytical data documentation (i.e., analysis data file or data package) is complete and includes sample preparation/extraction records, analysis sequence list, raw data,

calculations or calculation records, calibration data or records, QC measurement results, test results summary

- QC measurement results are within established program specification limits, or if not, the data are appropriately qualified
- Analytical sample holding times were met, or exceptions are documented

Independent technical review is required before any data are approved for release and submitted to the data reporting process. The independent technical review process is documented and archived in the associated data package/file.

SH 6.4 Data Reporting

After peer review of the data is completed as described in Section SH 6.3 the results are approved and a preliminary report is generated. The applicable Project Manager (PM) reviews the preliminary report (QAM Section 7.1.3), and works with necessary Analytical and Operations Sections' personnel to make any needed corrections, then a final report is produced. The final report (QAM Exhibit 7-2) is also reviewed and signed by the PM before it is submitted to the customer. Each final report has a unique identification number, which is the CTL Work Order No. listed in the upper right hand corner of the report.

The CTL laboratory offers four levels of data reports as illustrated in QAM Table 7-1. For the levels II, III, and IV deliverables formats, the applicable Analytical Sections provide the listed analysis portions and any related narrative comments to Project Management where the complete package is assembled. The PM reviews the complete package and writes the cumulative (i.e., case) analysis narrative. After final review, approval and signature by the PM, the report is paginated, copied, then mailed to the customer. The copy of the data package provided to the client and all associated raw data is kept for period of at least 3 years or longer if requested by the client. These records are stored in the laboratory for about six months then transferred to another company building for secure, long-term storage.

CTL provides electronic data deliverables (EDDs) in customer requested formats including Access, Excel, Quattro, dBaseIII+, and ASCII files. CTL's LIMS system stores information, from which the EDDs are prepared, for sample log-in, work status/tracking, sample results and associated QC results, report generation, and invoicing. CTL's Information System personnel maintain the LIMS on-site. Full server backups are performed nightly. Other electronic data include instrument magnetic tape media and are not overwritten. Backup copies of electronic media are prepared at least monthly and stored in a secure area off-site.

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Explosives by 8330**
Method: **EPA 8330**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,2-Dinitrobenzene	528-29-0	50	150	
Nitroguanidine	556-88-7	50	150	30

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Explosives by 8330**
Method: **EPA 8330B**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,2-Dinitrobenzene	528-29-0	81	117	
1,3,5-Trinitrobenzene	99-35-4	75	125	30
2,4,6-Trinitrotoluene	118-96-7	55	140	30
2,4-Dinitrotoluene	121-14-2	80	125	30
2,6-Dinitrotoluene	606-20-2	80	120	30
2-Amino-4,6-dinitrotoluene	35572-78-2	80	125	30
2-Nitrotoluene	88-72-2	80	125	30
3-Nitrotoluene	99-08-1	75	120	30
4-Amino-2,6-dinitrotoluene	19406-51-0	80	125	30
4-Nitrotoluene	99-99-0	75	125	30
HMX	2691-41-0	75	125	30
Nitrobenzene	98-95-3	75	125	30
Nitroglycerin	55-63-0	80	120	30
RDX	121-82-4	70	135	30
Tetryl	479-45-8	10	150	30

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Nitrocellulose**
Method: **EPA 9056M**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Nitrocellulose	9004-70-0	50	150	20

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Cyanide**
Method: **EPA 9012A**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Cyanide	57-12-5	69	128	20

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Mercury**
Method: **EPA 7471A**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Mercury	7439-97-6	80	120	20

CT LABORATORIES
delivering more than data from your environmental analyses

Test: **ICP Metals**
Method: **EPA 6010B**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Aluminum	7429-90-5	80	120	20
Antimony	7440-36-0	80	120	20
Arsenic	7440-38-2	80	120	20
Barium	7440-39-3	80	120	20
Beryllium	7440-41-7	80	120	20
Cadmium	7440-43-9	80	120	20
Calcium	7440-70-2	80	120	20
Chromium	7440-47-3	80	120	20
Cobalt	7440-48-4	80	120	20
Copper	7440-50-8	80	120	20
Iron	7439-89-6	80	120	20
Lead	7439-92-1	80	120	20
Magnesium	7439-95-4	80	120	20
Manganese	7439-96-5	80	120	20
Nickel	7440-02-0	80	120	20
Selenium	7782-49-2	80	120	20
Silver	7440-22-4	75	120	20
Thallium	7440-28-0	80	120	20
Vanadium	7440-62-2	80	120	20
Zinc	7440-66-6	80	120	20
Potassium	7440-09-7	80	120	20
Sodium	7440-23-5	80	120	20

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **PCB**
Method: **EPA 8082**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Aroclor-1016	12674-11-2	40	140	30
Aroclor-1260	11096-82-5	60	130	30
Surr: DCBP	2051-24-3	60	125	

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Pesticides**
Method: **EPA 8081A**

Matrix: **SOIL**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
4,4'-DDD	72-54-8	30	135	30
4,4'-DDE	72-55-9	70	125	30
4,4'-DDT	50-29-3	45	140	30
Aldrin	309-00-2	45	140	30
alpha-BHC	319-84-6	60	125	30
alpha-Chlordane	5103-71-9	65	120	30
beta-BHC	319-85-7	60	125	30
delta-BHC	319-86-8	55	130	30
Dieldrin	60-57-1	65	125	30
Endosulfan I	959-98-8	15	135	30
Endosulfan II	33213-65-9	35	140	30
Endosulfan sulfate	1031-07-8	60	135	30
Endrin	72-20-8	60	135	30
Endrin aldehyde	7421-93-4	35	145	30
Endrin ketone	53494-70-5	65	135	30
gamma-Chlordane	5103-74-2	65	125	30
Heptachlor	76-44-8	50	150	30
Heptachlor epoxide	1024-57-3	65	130	30
Lindane	58-89-9	60	125	30
Methoxychlor	72-43-5	55	145	30
SURR:2,4,5,6-CL4-m-xylene	877-09-8	70	125	
SURR:Decachlorobiphenyl	2051-24-3	55	130	

CT LABORATORIES
delivering more than data from your environmental analyses

Test: SVOC 8270 QSM
Method: EPA 8270C

Matrix: SOIL

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
2,4,5-Trichlorophenol	95-95-4	50	110	30
2,4,6-Trichlorophenol	88-06-2	45	110	30
2,4-Dichlorophenol	120-83-2	45	110	30
2,4-Dimethylphenol	105-67-9	30	105	30
2,4-Dinitrophenol	51-28-5	15	130	30
2,4-Dinitrotoluene	121-14-2	50	115	30
2,6-Dinitrotoluene	606-20-2	50	110	30
2-Chloronaphthalene	91-58-7	45	105	30
2-Chlorophenol	95-57-8	45	105	30
2-Methylnaphthalene	91-57-6	45	105	30
2-Methylphenol	95-48-7	40	105	30
2-Nitroaniline	88-74-4	45	120	30
2-Nitrophenol	88-75-5	40	110	30
3 & 4-Methylphenol	1319-77-3	40	105	30
3,3'-Dichlorobenzidine	91-94-1	10	130	30
3-Nitroaniline	99-09-2	25	110	30
4,6-Dinitro-2-methylphenol	534-52-1	30	135	30
4-Bromophenyl-phenyl ether	101-55-3	45	115	30
4-Chloro-3-methylphenol	59-50-7	45	115	30
4-Chloroaniline	106-47-8	10	95	30
4-Chlorophenyl-phenyl ether	7005-72-3	45	110	30
4-Nitroaniline	100-01-6	35	115	30
4-Nitrophenol	100-02-7	15	140	30
Acenaphthene	83-32-9	45	110	30
Acenaphthylene	208-96-8	45	105	30
Anthracene	120-12-7	55	105	30
Benzo(a)anthracene	56-55-3	50	110	30
Benzo(a)pyrene	50-32-8	50	110	30
Benzo(b)fluoranthene	205-99-2	45	115	30
Benzo(g,h,i)perylene	191-24-2	40	125	30
Benzo(k)fluoranthene	207-08-9	45	125	30
Bis(2-chloroethoxy)methane	111-91-1	45	110	30
Bis(2-chloroethyl)ether	111-44-4	40	105	30
Bis(2-ethylhexyl)phthalate	117-81-7	45	125	30
Butylbenzylphthalate	85-68-7	50	125	30
Carbazole	86-74-8	45	115	30
Chrysene	218-01-9	55	110	30
Di-n-butylphthalate	84-74-2	55	110	30
Di-n-octylphthalate	117-84-0	40	130	30
Dibenzo(a,h)anthracene	53-70-3	40	125	30
Dibenzofuran	132-64-9	50	105	30
Diethylphthalate	84-66-2	50	115	30
Dimethylphthalate	131-11-3	50	110	30
Fluoranthene	206-44-0	55	115	30
Fluorene	86-73-7	50	110	30
Hexachlorobenzene	118-74-1	45	120	30
Hexachlorobutadiene	87-68-3	40	115	30
Hexachlorocyclopentadiene	77-47-4	30	137	30
Hexachloroethane	67-72-1	35	110	30
Indeno(1,2,3-cd)pyrene	193-39-5	40	120	30
Isophorone	78-59-1	45	110	30
N-Nitroso-di-n-propylamine	621-64-7	40	115	30
N-Nitrosodiphenylamine & Diphn	86-30-6/122-39-4	50	115	30
Naphthalene	91-20-3	40	105	30
Nitrobenzene	98-95-3	40	115	30
Pentachlorophenol	87-86-5	25	120	30
Phenanthrene	85-01-8	50	110	30
Phenol	108-95-2	40	100	30
Pyrene	129-00-0	45	125	30
Surr: 2,4,6-Tribromophenol	118-79-6	35	125	
Surr: 2-Fluorobiphenyl	321-60-8	45	105	
Surr: 2-Fluorophenol	367-12-4	35	105	
Surr: Nitrobenzene-d5	4165-60-0	35	100	
Surr: Phenol-d5	4165-62-2	40	100	
Surr: Terphenyl-d14	98904-43-9	30	125	

CT LABORATORIES

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Test: VOC by 8260
Method: EPA 8260B

Matrix: SOIL

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,1,1-Trichloroethane	71-55-6	70	135	30
1,1,2,2-Tetrachloroethane	79-34-5	55	130	30
1,1,2-Trichloroethane	79-00-5	60	125	30
1,1-Dichloroethane	75-34-3	75	125	30
1,1-Dichloroethene	75-35-4	65	135	30
1,2 Dichloroethane-d4	17060-07-0	70	130	
1,2,3-Trichlorobenzene	87-61-6	60	135	30
1,2,4-Trichlorobenzene	120-82-1	65	130	30
1,2-Dibromo-3-chloropropane	96-12-8	40	135	30
1,2-Dibromoethane	106-93-4	70	125	30
1,2-Dichlorobenzene	95-50-1	75	120	30
1,2-Dichloroethane	107-06-2	70	135	30
1,2-Dichloropropane	78-87-5	70	120	30
1,3-Dichlorobenzene	541-73-1	70	125	30
1,4-Dichlorobenzene	106-46-7	70	125	30
112Trichloro122trifluoroethane	76-13-1	70	130	30
2-Butanone	78-93-3	30	160	30
2-Hexanone	591-78-6	45	145	30
4-Methyl-2-pentanone	108-10-1	20	145	30
Acetone	67-64-1	20	160	30
Benzene	71-43-2	75	125	30
Bromochloromethane	74-97-5	70	125	30
Bromodichloromethane	75-27-4	70	130	30
Bromofluorobenzene	460-00-4	85	120	
Bromoform	75-25-2	55	135	30
Bromomethane	74-83-9	30	160	30
Carbon disulfide	75-15-0	45	160	30
Carbon tetrachloride	56-23-5	65	135	30
Chlorobenzene	108-90-7	75	125	30
Chloroethane	75-00-3	40	155	30
Chloroform	67-66-3	70	125	30
Chloromethane	74-87-3	50	130	30
cis-1,2-Dichloroethene	156-59-2	65	125	30
cis-1,3-Dichloropropene	10061-01-5	70	125	30
d8-Toluene	2037-26-5	85	115	
Dibromochloromethane	124-48-1	65	130	30
Dibromofluoromethane	1868-53-7	70	130	
Dichlorodifluoromethane	75-71-8	35	135	30
Ethylbenzene	100-41-4	75	125	30
Isopropylbenzene	98-82-8	75	130	30
m & p-Xylene	1330-20-7	80	125	30
Methyl tert-butyl ether	1634-04-4	55	140	30
Methylene chloride	75-09-2	55	140	30
o-Xylene	95-47-6	75	125	30
Styrene	100-42-5	75	125	30
Tetrachloroethene	127-18-4	65	140	30
Toluene	108-88-3	70	125	30
trans-1,2-Dichloroethene	156-60-5	65	135	30
trans-1,3-Dichloropropene	10061-02-6	65	125	30
Trichloroethene	79-01-6	75	125	30
Trichlorofluoromethane	75-69-4	25	185	30
Vinyl chloride	75-01-4	60	125	30

CT LABORATORIES

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Test: **Explosives by 8330**
Method: **EPA 8330**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,2-Dinitrobenzene	528-29-0	50	150	
Nitroguanidine	556-88-7	50	150	30

CT LABORATORIES

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Test: **Explosives by 8330**
Method: **EPA 8330B**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,2-Dinitrobenzene	528-29-0	50	150	30
1,3,5-Trinitrobenzene	99-35-4	65	140	30
2,4,6-Trinitrotoluene	118-96-7	50	145	30
2,4-Dinitrotoluene	121-14-2	60	135	30
2,6-Dinitrotoluene	606-20-2	60	135	30
2-Amino-4,6-dinitrotoluene	35572-78-2	50	155	30
2-Nitrotoluene	88-72-2	45	135	30
3-Nitrotoluene	99-08-1	50	130	30
4-Amino-2,6-dinitrotoluene	19406-51-0	55	155	30
4-Nitrotoluene	99-99-0	50	130	30
HMX	2691-41-0	80	115	30
Nitrobenzene	98-95-3	50	140	30
Nitroglycerin	55-63-0	50	150	30
RDX	121-82-4	50	160	30
Tetryl	479-45-8	20	175	30

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Test: **Nitrocellulose**
Method: **EPA 9056M**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Nitrocellulose	9004-70-0	50	150	30

CT LABORATORIES

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Test: **Anions, Total QSM**
Method: **EPA 9056**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Nitrate Nitrogen Total	14797-55-8	80	120	20
Nitrite Nitrogen Total	14797-65-0	80	120	20

CT LABORATORIES

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Test: **Cyanide**
Method: **EPA 9012A**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Cyanide	57-12-5	69	133	20

CT LABORATORIES

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Test: **Mercury, Dissolved**
Method: **EPA 7470**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Dissolved Mercury	7439-97-6	80	120	20

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Mercury, Total**
Method: **EPA 7470**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Total Mercury	7439-97-6	80	120	20

CT LABORATORIES
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Test: **ICP Metals, Dissolved**
Method: **EPA 6010B**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Dissolved Aluminum	7429-90-5	80	120	20
Dissolved Antimony	7440-36-0	80	120	20
Dissolved Arsenic	7440-38-2	80	120	20
Dissolved Barium	7440-39-3	80	120	20
Dissolved Beryllium	7440-41-7	80	120	20
Dissolved Cadmium	7440-43-9	80	120	20
Dissolved Calcium	7440-70-2	80	120	20
Dissolved Chromium	7440-47-3	80	120	20
Dissolved Cobalt	7440-48-4	80	120	20
Dissolved Copper	7440-50-8	80	120	20
Dissolved Iron	7439-89-6	80	120	20
Dissolved Lead	7439-92-1	80	120	20
Dissolved Magnesium	7439-95-4	80	120	20
Dissolved Manganese	7439-96-5	80	120	20
Dissolved Nickel	7440-02-0	80	120	20
Dissolved Selenium	7782-49-2	80	120	20
Dissolved Silver	7440-22-4	80	120	20
Dissolved Thallium	7440-28-0	80	120	20
Dissolved Vanadium	7440-62-2	80	120	20
Dissolved Zinc	7440-66-6	80	120	20
Dissolved Sodium	7440-23-5	80	120	20
Dissolved Potassium	7440-09-7	80	120	20

CT LABORATORIES
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Test: **ICP Metals, Total**
Method: **EPA 6010B**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Total Aluminum	7429-90-5	80	120	20
Total Antimony	7440-36-0	80	120	20
Total Arsenic	7440-38-2	80	120	20
Total Barium	7440-39-3	80	120	20
Total Beryllium	7440-41-7	80	120	20
Total Cadmium	7440-43-9	80	120	20
Total Calcium	7440-70-2	80	120	20
Total Chromium	7440-47-3	80	120	20
Total Cobalt	7440-48-4	80	120	20
Total Copper	7440-50-8	80	120	20
Total Iron	7439-89-6	80	120	20
Total Lead	7439-92-1	80	120	20
Total Magnesium	7439-95-4	80	120	20
Total Manganese	7439-96-5	80	120	20
Total Nickel	7440-02-0	80	120	20
Total Selenium	7782-49-2	80	120	20
Total Silver	7440-22-4	80	120	20
Total Thallium	7440-28-0	80	120	20
Total Vanadium	7440-62-2	80	120	20
Total Zinc	7440-66-6	80	120	20
Total Potassium	7440-09-7	80	120	20
Total Sodium	7440-23-5	80	120	20

CT LABORATORIES

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Test: **PCB**
Method: **EPA 8082**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Aroclor-1016	12674-11-2	25	145	30
Aroclor-1260	11096-82-5	30	145	30
Surr: DCBP	2051-24-3	30	135	

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Pesticides**
Method: **EPA 8081A**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
4,4'-DDD	72-54-8	25	150	30
4,4'-DDE	72-55-9	35	140	30
4,4'-DDT	50-29-3	45	140	30
Aldrin	309-00-2	25	140	30
alpha-BHC	319-84-6	60	130	30
alpha-Chlordane	5103-71-9	65	125	30
beta-BHC	319-85-7	65	125	30
delta-BHC	319-86-8	45	135	30
Dieldrin	60-57-1	60	130	30
Endosulfan I	959-98-8	50	110	30
Endosulfan II	33213-65-9	30	130	30
Endosulfan sulfate	1031-07-8	55	135	30
Endrin	72-20-8	55	135	30
Endrin aldehyde	7421-93-4	55	135	30
Endrin ketone	53494-70-5	75	125	30
gamma-Chlordane	5103-74-2	60	125	30
Heptachlor	76-44-8	40	130	30
Heptachlor epoxide	1024-57-3	60	130	30
Lindane	58-89-9	25	135	30
Methoxychlor	72-43-5	55	150	30
SURR:2,4,5,6-CL4-m-xylene	877-09-8	25	140	
SURR:Decachlorobiphenyl	2051-24-3	30	135	

CT LABORATORIES
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Test: SVOC 8270 QSM
Method: EPA 8270C

Matrix: WATER

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
2,4,5-Trichlorophenol	95-95-4	50	110	30
2,4,6-Trichlorophenol	88-06-2	50	115	30
2,4-Dichlorophenol	120-83-2	50	105	30
2,4-Dimethylphenol	105-67-9	30	110	30
2,4-Dinitrophenol	51-28-5	15	140	30
2,4-Dinitrotoluene	121-14-2	50	120	30
2,6-Dinitrotoluene	606-20-2	50	115	30
2-Chloronaphthalene	91-58-7	50	105	30
2-Chlorophenol	95-57-8	35	105	30
2-Methylnaphthalene	91-57-6	45	105	30
2-Methylphenol	95-48-7	40	110	30
2-Nitroaniline	88-74-4	50	115	30
2-Nitrophenol	88-75-5	40	115	30
3 & 4-Methylphenol	1319-77-3	30	110	30
3,3'-Dichlorobenzidine	91-94-1	20	110	30
3-Nitroaniline	99-09-2	20	125	30
4,6-Dinitro-2-methylphenol	534-52-1	40	130	30
4-Bromophenyl-phenyl ether	101-55-3	50	115	30
4-Chloro-3-methylphenol	59-50-7	45	110	30
4-Chloroaniline	106-47-8	15	110	30
4-Chlorophenyl-phenyl ether	7005-72-3	50	110	30
4-Nitroaniline	100-01-6	35	120	30
4-Nitrophenol	100-02-7	1	125	30
Acenaphthene	83-32-9	45	110	30
Acenaphthylene	208-96-8	50	105	30
Anthracene	120-12-7	55	110	30
Benzo(a)anthracene	56-55-3	55	110	30
Benzo(a)pyrene	50-32-8	55	110	30
Benzo(b)fluoranthene	205-99-2	45	120	30
Benzo(g,h,i)perylene	191-24-2	40	125	30
Benzo(k)fluoranthene	207-08-9	45	125	30
Bis(2-chloroethoxy)methane	111-91-1	45	105	30
Bis(2-chloroethyl)ether	111-44-4	35	110	30
Bis(2-ethylhexyl)phthalate	117-81-7	40	125	30
Butylbenzylphthalate	85-68-7	45	115	30
Carbazole	86-74-8	50	115	30
Chrysene	218-01-9	55	110	30
Di-n-butylphthalate	84-74-2	55	115	30
Di-n-octylphthalate	117-84-0	35	115	30
Dibenzo(a,h)anthracene	53-70-3	40	125	30
Dibenzofuran	132-64-9	55	105	30
Diethylphthalate	84-66-2	40	120	30
Dimethylphthalate	131-11-3	25	125	30
Fluoranthene	206-44-0	55	115	30
Fluorene	86-73-7	50	110	30
Hexachlorobenzene	118-74-1	50	110	30
Hexachlorobutadiene	87-68-3	25	105	30
Hexachlorocyclopentadiene	77-47-4	36	106	30
Hexachloroethane	67-72-1	30	95	30
Indeno(1,2,3-cd)pyrene	193-39-5	45	125	30
Isophorone	78-59-1	50	110	30
N-Nitroso-di-n-propylamine	621-64-7	35	130	30
N-Nitrosodiphenylamine & Diphn	86-30-6/122-39-4	50	110	30
Naphthalene	91-20-3	40	100	30
Nitrobenzene	98-95-3	45	110	30
Pentachlorophenol	87-86-5	40	115	30
Phenanthrene	85-01-8	50	115	30
Phenol	108-95-2	1	115	30
Pyrene	129-00-0	50	130	30
Surr: 2,4,6-Tribromophenol	118-79-6	40	125	30
Surr: 2-Fluorobiphenyl	321-60-8	50	110	30
Surr: 2-Fluorophenol	367-12-4	20	110	30
Surr: Nitrobenzene-d5	4165-60-0	40	110	30
Surr: Phenol-d5	4165-62-2	10	115	30
Surr: Terphenyl-d14	98904-43-9	50	135	30

CT LABORATORIES
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Test: SVOC 8270 + PAH SIM
Method: EPA 8270C

Matrix: WATER

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
2,4,5-Trichlorophenol	95-95-4	45	135	40
2,4,6-Trichlorophenol	88-06-2	45	135	40
2,4-Dichlorophenol	120-83-2	45	135	40
2,4-Dimethylphenol	105-67-9	45	135	40
2,4-Dinitrophenol	51-28-5	45	135	40
2,4-Dinitrotoluene	121-14-2	45	135	40
2,6-Dinitrotoluene	606-20-2	45	135	40
2-Chloronaphthalene	91-58-7	45	135	40
2-Chlorophenol	95-57-8	45	135	40
2-Methylnaphthalene	91-57-6	45	135	40
2-Methylphenol	95-48-7	45	135	40
2-Nitroaniline	88-74-4	45	135	40
2-Nitrophenol	88-75-5	45	135	40
3 & 4-Methylphenol	1319-77-3	45	135	40
3,3'-Dichlorobenzidine	91-94-1	45	135	40
3-Nitroaniline	99-09-2	45	135	40
4,6-Dinitro-2-methylphenol	534-52-1	45	135	40
4-Bromophenyl-phenyl ether	101-55-3	45	135	40
4-Chloro-3-methylphenol	59-50-7	45	135	40
4-Chloroaniline	106-47-8	45	135	40
4-Chlorophenyl-phenyl ether	7005-72-3	45	135	40
4-Nitroaniline	100-01-6	45	135	40
4-Nitrophenol	100-02-7	45	135	40
Acenaphthene	83-32-9	45	135	40
Acenaphthylene	208-96-8	45	135	40
Anthracene	120-12-7	45	135	40
Benzo(a)anthracene	56-55-3	45	135	40
Benzo(a)pyrene	50-32-8	45	135	40
Benzo(b)fluoranthene	205-99-2	45	135	40
Benzo(g,h,i)perylene	191-24-2	45	135	40
Benzo(k)fluoranthene	207-08-9	45	135	40
Bis(2-chloroethoxy)methane	111-91-1	45	135	40
Bis(2-chloroethyl)ether	111-44-4	45	135	40
Bis(2-ethylhexyl)phthalate	117-81-7	45	135	40
Butylbenzylphthalate	85-68-7	45	135	40
Carbazole	86-74-8	45	135	40
Chrysene	218-01-9	45	135	40
Di-n-butylphthalate	84-74-2	45	135	40
Di-n-octylphthalate	117-84-0	45	135	40
Dibenzo(a,h)anthracene	53-70-3	45	135	40
Dibenzofuran	132-64-9	45	135	40
Diethylphthalate	84-66-2	45	135	40
Dimethylphthalate	131-11-3	45	135	40
Fluoranthene	206-44-0	45	135	40
Fluorene	86-73-7	45	135	40
Hexachlorobenzene	118-74-1	45	135	40
Hexachlorobutadiene	87-68-3	45	135	40
Hexachlorocyclopentadiene	77-47-4	45	135	40
Hexachloroethane	67-72-1	45	135	40
Indeno(1,2,3-cd)pyrene	193-39-5	45	135	40
Isophorone	78-59-1	45	135	40
N-Nitroso-di-n-propylamine	621-64-7	45	135	40
N-Nitrosodiphenylamine & Diphn	86-30-6/122-39-4	45	135	40
Naphthalene	91-20-3	45	135	40
Nitrobenzene	98-95-3	45	135	40
Pentachlorophenol	87-86-5	45	135	40
Phenanthrene	85-01-8	45	135	40
Phenol	108-95-2	45	135	40
Pyrene	129-00-0	45	135	40
Surr: 2,4,6-Tribromophenol	118-79-6	50	150	
Surr: 2-Fluorobiphenyl	321-60-8	50	150	
Surr: 2-Fluorophenol	367-12-4	50	150	
Surr: Nitrobenzene-d5	4165-60-0	50	150	
Surr: o-terphenyl-d14	5142-67-6	50	150	
Surr: Phenol-d5	4165-62-2	50	150	
Surr: Terphenyl-d14	98904-43-9	50	150	

CT LABORATORIES

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Test: **Organic Carbon, Total**
Method: **EPA 9060**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Total Organic Carbon	TOC	84	114	16

CT LABORATORIES

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Test: **Volatile Organic Compounds 8260**
Method: **EPA 8260B**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
1,1,1-Trichloroethane	71-55-6	65	130	20
1,1,2,2-Tetrachloroethane	79-34-5	65	130	20
1,1,2-Trichloroethane	79-00-5	75	125	20
1,1-Dichloroethane	75-34-3	70	135	20
1,1-Dichloroethene	75-35-4	70	130	20
1,2 Dichloroethane-d4	17060-07-0	70	120	
1,2,3-Trichlorobenzene	87-61-6	55	140	20
1,2,4-Trichlorobenzene	120-82-1	65	135	20
1,2-Dibromo-3-chloropropane	96-12-8	50	130	20
1,2-Dibromoethane	106-93-4	80	120	20
1,2-Dichlorobenzene	95-50-1	70	120	20
1,2-Dichloroethane	107-06-2	70	130	20
1,2-Dichloropropane	78-87-5	75	125	20
1,3-Dichlorobenzene	541-73-1	75	125	20
1,4-Dichlorobenzene	106-46-7	75	125	20
112Trichloro122trifluoroethane	76-13-1	70	130	20
2-Butanone	78-93-3	30	150	20
2-Hexanone	591-78-6	55	130	20
4-Methyl-2-pentanone	108-10-1	60	135	20
Acetone	67-64-1	40	140	20
Benzene	71-43-2	80	120	20
Bromochloromethane	74-97-5	65	130	20
Bromodichloromethane	75-27-4	75	120	20
Bromofluorobenzene	460-00-4	75	120	
Bromoform	75-25-2	70	130	20
Bromomethane	74-83-9	30	145	20
Carbon disulfide	75-15-0	35	160	20
Carbon tetrachloride	56-23-5	65	140	20
Chlorobenzene	108-90-7	80	120	20
Chloroethane	75-00-3	60	135	20
Chloroform	67-66-3	65	135	20
Chloromethane	74-87-3	40	125	20
cis-1,2-Dichloroethene	156-59-2	70	125	20
cis-1,3-Dichloropropene	10061-01-5	70	130	20
d8-Toluene	2037-26-5	85	120	
Dibromochloromethane	124-48-1	60	135	20
Dibromofluoromethane	1868-53-7	85	115	
Dichlorodifluoromethane	75-71-8	30	155	20
Ethylbenzene	100-41-4	75	125	20
Isopropylbenzene	98-82-8	75	125	20
m & p-Xylene	1330-20-7	75	130	20
Methyl tert-butyl ether	1634-04-4	65	125	20
Methylene chloride	75-09-2	55	140	20
o-Xylene	95-47-6	80	120	20
Styrene	100-42-5	65	135	20
Tetrachloroethene	127-18-4	45	150	20
Toluene	108-88-3	75	120	20
trans-1,2-Dichloroethene	156-60-5	60	140	20
trans-1,3-Dichloropropene	10061-02-6	55	140	20
Trichloroethene	79-01-6	70	125	20
Trichlorofluoromethane	75-69-4	60	145	20
Vinyl chloride	75-01-4	50	145	20

CT LABORATORIES

delivering more than data from your environmental analyses

Test: **Perchlorate 6850**
Method: **EPA 6850**

Matrix: **WATER**

Analyte	CAS #	LCL % Recovery	UCL % Recovery	RPD Limit
Perchlorate	14797-73-0	80	120	15

ATTACHMENT 3
Field Sampling Standard Operating Procedures (SOPs)

Earth Resources Technology, Inc. Standard Operating Procedure **PREPARING FOR AND CONCLUDING FIELD ACTIVITY**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-01-1A**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
Signature	1/23/09 Date	Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to outline requirements associated with preparing for and concluding environmental field activities.

2.0 SCOPE

This procedure applies to all field activities commencing with the Notice to Proceed and ending with the completion of all project field activities.

It is neither the intent of this procedure to fully detail all actions required for preparing and concluding field activities nor define specific methodology, but rather offer general points to be considered in the preparation and conclusion of field activities. Preparation and conclusion activities are specific to the planned field activities and will be detailed in the project-specific Sample and Analysis Plan.

3.0 REQUIREMENTS

In order to efficiently complete field work, specific tasks must be accomplished in an orderly fashion prior to actual field work (preparation) and after field work has been completed (conclusion).

4.0 RESPONSIBILITIES

4.1 Project Manager

For the purpose of this procedure, the Project Manager is responsible for providing the Field Team access to applicable project-specific information, subcontractor support and equipment necessary to complete stated Project Quality Objectives. Project Manager responsibilities include, but are not limited to, locating resources and manufacturers of the proposed equipment, initiating purchaser lease agreements, coordinating site access, coordinating field tasks with associated subcontractors/prime contractors and perform overall task management.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring the Field Team understands all associated field activities and applicable procedures, adheres to the project-specific work plans and successfully completes the field activities. The Field Team Leader is responsible for identifying and obtaining project specific field equipment and tools, appropriate field recording forms and ensuring adequate quantities of supplies. If appropriate, the Field Team Leader is responsible for coordinating necessary subcontractor support. This may include scheduling the receipt of appropriate environmental sample containers, scheduling courier services for analytical samples to the laboratory, etc, as directed by the Project Manager.

The Field Team Leader is responsible for the completion of the Field Activity Preparation Checklist and the Work Order Form. Upon completion, these forms will be reviewed with the Project Manager to ensure accuracy and completeness.

During field activities and at the conclusion of field activities, the Field Team Leader is responsible for performing periodic quality control and quality assurance checks of all environmental sampling procedures and samples obtained, arrange sample shipment to the analytical laboratory, ensure all equipment (both rented and ERT owned) and supplies are accounted for and aptly returned.

4.3 Field Team Members

The Field Team is responsible for the successful completion of all field tasks as assigned by the Field Team Leader. This will be ensured by the adherence to the project-specific operating procedure and project-specific work plan(s). For the purpose of this procedure, the Field Team will be required to fully understand tasks associated with the project-specific work plan(s). During preparation for field activities, field team members may be directed by the Field Team Leader to aid in the identification and arrangement of necessary activity specific equipment, subcontractor support and information.

5.0 EQUIPMENT

None Specified

6.0 PROCEDURE

6.1 Preparing Field Activities

Preparation is a process that begins with the Notice to Proceed and ends with the initiation of field activities. Adequate time and effort involved in preparation ensures efficient and effective execution of the work plan and completion of field activities.

The many preparation tasks to be considered include:

- Ordering and procuring items of a specialized nature, including environmental sampling equipment, environmental sample containers, etc.
- Performing a thorough review of the cost proposal to determine if additional items may be needed. This should be discussed with the personnel assigned to field activities.
- Informing personnel of the date, location and activity required to be performed. Instruct personnel as to travel arrangements.
- Locating sources for field purchased items and supplies.
- Establishing an inventory system of disposable and non-disposable items.
- Detailing specific requirements for mobilization of subcontractors to include drilling contractors, analytical laboratories. Activities to be considered include transportation, decontamination, orientation and badging, and initial setup.
- Testing and calibrating all equipment to ensure operational readiness.
- Establishing a field office or field staging areas for materials and IDW.
- Ensuring reliable communications for field personnel during field activities.

6.2 Concluding Field Activities

Terminating field activity includes activities necessary to transfer custody of materials and supplies after completion of field activities. Activities to be considered include:

- Planning for the thorough completion of field activities before conclusion of field activities. Preliminary conclusion efforts may be undertaken, but all materials and supplies necessary for field activities shall be retained until field activities are complete.
- Reviewing records and thoroughly inspecting equipment to ensure all equipment has been decontaminated.
- Adequately packing special equipment, electronic equipment and other non-disposable items for shipment.
- Completing a review of all environmental samples to be delivered to the analytical laboratory. All sample volumes, sample nomenclature, sample labels, specified sample analytics, number of samples, Chain-of Custody forms, packaging of samples and custody seal will be thoroughly reviewed by the Field Team Leader prior to relinquishing environmental samples.
- Completing an inventory review of packaged equipment for shipment.
- Contact applicable rental equipment suppliers, off-rent equipment and document confirmation off-rent number when applicable. Transfer custody of the rental equipment to the rental company or contracted courier.
- Stage and or dispose of all IDW in accordance with federal and state regulations

7.0 REFERENCES

The following documents were referenced during the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

None

9.0 ATTACHMENTS

9.1 Attachment 1 – ERT Field Activity Preparation Checklist

Prior to commencing field activities, the field team is required to amass the necessary information and equipment necessary to successfully complete the field activity objectives. To ensure the appropriate equipment has been obtained, the Field Activity Preparation Checklist will be completed and reviewed with the Field Team

ERT Field Activity Preparation Checklist

General:

- ☐ Relevant project file documents
- ☐ Field book
- ☐ Clip board / desk top
- ☐ Cell phone and charger
- ☐ Safety glasses
- ☐ Steel toe boots
- ☐ Hard hat
- ☐ Work plan
- ☐ Work gloves
- ☐ Hand tools
- ☐ HASP
- ☐ Paper towels
- ☐ Wide brimmed hat
- ☐ Caution tape
- ☐ Nitrile gloves
- ☐ Trash bags
- ☐ Chair and table
- ☐ Lunch
- ☐ Drinking water
- ☐ Rugged work ware
- ☐ Rain Gear
- ☐ Site access key
- ☐ Subcontractor contact #
- ☐ Power converter

Drilling

- ☐ PID w/ cal gas
- ☐ Water level meter
- ☐ GPS
- ☐ Decon bucket, soap and brush
- ☐ Well log forms
- ☐ Work plan
- ☐ Caution tape
- ☐ Measuring wheel
- ☐ USACE manual
- ☐ Macro core holder
- ☐ Non-HAZ stickers
- ☐ Hand auger

Ground water Sampling

- ☐ Pump
- ☐ Battery / Generator
- ☐ Tubing
- ☐ Multi-meter
- ☐ Bailers
- ☐ Measuring cup
- ☐ Stopwatch
- ☐ PID w/ cal gas
- ☐ Buckets (3) w/ lids
- ☐ Decon station
- ☐ Soap and brush
- ☐ Distilled water
- ☐ DI water
- ☐ Water level meter
- ☐ String
- ☐ Well keys
- ☐ Carbon bucket
- ☐ Bottle ware + extras
- ☐ Labels + extras
- ☐ COC
- ☐ Cooler with bubble wrap
- ☐ Ice
- ☐ Ziplock bags
- ☐ Fishing gear
- ☐ Work plan
- ☐ Work order
- ☐ Purge data sheets
- ☐ Non-HAZ stickers

Soil Sampling

- ☐ PID w/ cal gas
- ☐ zip lock bags
- ☐ Jars
- ☐ Cooler and bubble wrap
- ☐ Ice
- ☐ Chain of custody
- ☐ Labels
- ☐ Soil sample equipment
- ☐ Hand auger
- ☐ Digging Bar
- ☐ Paper towels
- ☐ Trowel

**Highlight as applicable*

Earth Resources Technology, Inc. Standard Operating Procedure **USE OF FIELD LOGBOOKS**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-01-2**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/22/09 Date	 Signature	1/21/09 Date

1.0 PURPOSE

The purpose of this procedure is to detail minimum requirements related to maintaining Field Logbooks and recording all field activities within Field Logbooks.

2.0 SCOPE

This procedure applies to all Field Logbooks which are required to be maintained onsite during field activities and for the duration of a project.

3.0 REQUIREMENTS

Field Logbooks will be initiated at the start of the first field activity. Entries will be made each day that field activities occur related to Earth Resources Technology, Inc. (ERT) or ERT subcontractor activities. A current field logbook will be maintained during the duration of the project. Site activities which are nonconcurring will be recorded in the same dedicated Field Logbook.

The Field Logbook will become part of the permanent project file. Field Logbooks must be maintained properly and to the standard set forth in this procedure because information contained in the Field Logbook may be admitted as evidence in mitigation, as an accurate record of field procedures and/or representative site conditions during the time of field activities. All logbooks will be secured in a reasonable fashion for the duration of the project and filed with related project documents after the completion of the project. Field Logbooks are considered company property, as such will be retained and utilized by ERT personnel exclusively to record the events of field activities associated with ERT projects.

4.0 RESPONSIBILITIES

4.1 Project Managers

The Field Logbook is issued by the Project Manager to the Field Team Leader or other person responsible for the direction of field activities (i.e., Field Geologist, Sampling Team Leader). Upon completing field activities, the Field Logbook will be returned to the Project Manager's custody that is responsible for reviewing the daily entries and filing the logbook within the permanent project file.

4.2 Field Team Leader

Field Logbooks are issued to the Field Team Leader, or other person responsible for conducting field activities. It is the responsibility of this person to keep the logbook current, detailing all field activities and pertinent information. It is this person's responsibility to properly secure the logbook and return the logbook to the custody of the Project Manager after concluding field efforts.

5.0 EQUIPMENT

The Field Logbook shall have pre-printed numbered pages, be bound in such a way that pages cannot be readily removed, and be constructed of robust and weather resistant material.

6.0 PROCEDURE

6.1 General

The cover and inside cover of each logbook will contain at a minimum the following information:

- Project Identification
- Project number
- Project Manager's name
- Project Manager contact info
- Sequential book number
- Start date
- End date

The spine of the log book shall contain the following information

- Project Identification
- Sequential book number

As appropriate, a table of contents will be compiled on the Table of Contents page with descriptions of field activities and their respective page numbers in chronological order. The Tables of Contents page will be completed after a log book has been filled to capacity and review of the daily entries has been performed by the Project Manager.

Unless prohibited by weather, pens with permanent black ink should be used to record all activities and datum. When weather conditions do not make it conducive to use permanent ink, entries should be made using a non-smear lead pencil. Once removed from wet elements, penciled entries should be repeated with a permanent ink pen to ensure permanency of the entry. No erasures are permitted. Data or other information that has been entered incorrectly will be corrected by drawing a single line through the incorrect entry, initialing (or signing) and dating the lined-through entry. Under no circumstances will the incorrect material be erased, made illegible or obscured so that it cannot be read. The Field Team Leader or his designee will draw a diagonal line and initial at 1) the end of unfilled pages and 2) the end of all entries for each day of field activity. The final recorded information for each daily entry will be the time field personnel exited the site.

6.2 Information Required

The initial entry for each day of field activities at a minimum should include the following information:

- Date
- Day of week
- Purpose of site visit
- Time of arrival onsite
- Weather conditions
- Names and/or initials of all ERT field personnel present
- Names and/or initials of all subcontractors present
- Names of any visitors present and their affiliation

Entries will be recorded in the Field Logbook in real-time chronological order and summarize all site activities. Logbook entries will be recorded in clear concise and legible hand script, should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate.

6.2.1 Information Required for Sampling Activities

- Makes and models of equipment, and identifying numbers
- Equipment calibration procedures performed including concentrations of calibration media
- Equipment decontamination procedures performed
- Equipment nonconformance
- Sampling methodology utilized
- Matrix sampled
- Sample location, when applicable
- Sample ID's
- Sample collection times
- Sample ware, i.e. number of, size, type, and preservative
- Analytical parameters requested to be performed by the contracted laboratory
- Sample custody procedure conducted
- Any deviations from the work plan that occur while conducting field activities
- Relevant Health and Safety conditions
- Notation of use of any activity specific forms utilized

6.2.2 Information Required for Soil Boring and Well Installation

- Name of subcontractor(s)
- Names of the subcontractor(s) personnel
- Drilling methodology utilized
- Location of drilling activities and the duration of drilling (start and completion times).

- Observations made during the recovery of boring cuttings, including visual, olfactory, photoioinzation detector (PID) readings, volume and geologic descriptors, if necessary.
- Well construction information, including the length of screen and riser, depth interval of filter pack, bentonite, and sealing media, depth of screened interval, and well head protection measures.
- Quantity, type, and name of manufacture of well construction material (i.e. filter sand, bentonite, grout, and well material)
- Quantity and frequency that water was added to the borehole in order to assist boring and well construction
- Depth groundwater was encountered, as applicable.
- Depth to water in completed well.
- Any complications impeding the progress of drilling activities
- Abandonment method if no well is installed in borehole
- Site sketch depicting relevant surface features, project related investigative features, and scale and North arrow if possible.
- Any deviations from the work plan that occur while conducting field activities
- Relevant Health and Safety conditions
- Notation of use of any activity specific forms utilized

Field Logbook entries are not intended to replace data recorded on activity specific data forms such as Well Log Forms, Well Construction Diagram Forms, and Purge Data Sheets. Use of such forms must be noted in the Field Logbook.

7.0 REFERENCES

The following documents were referenced in the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 2007. *Field Branches Quality System and Technical Procedures*. Region 4 Science and Ecosystem Support Division. SESDPROC-010-R3. November

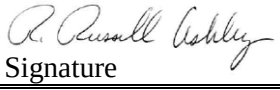
Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York.

8.0 DEFINITIONS

None

9.0 ATTACHMENTS

None

Earth Resources Technology, Inc. Standard Operating Procedure **DECONTAMINATION OF SAMPLING EQUIPMENT**			
Effective Date: **01/22/09**	Version: **001**	**ERT SOP-03-1**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/23/09 Date	 Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide reference information on the proper decontamination procedures for sampling equipment used to perform field investigations.

2.0 SCOPE

This procedure addresses decontamination of sampling equipment. Decontamination of drilling equipment and other well installation equipment is described in ERT SOP-03-2.

3.0 REQUIREMENTS

To ensure that chemical analysis results are reflective of actual constituent concentrations present at sampling locations and to minimize the potential for introducing foreign constituents, equipment used in sampling activities must be properly cleaned and decontaminated. This minimizes or eliminates the potential for cross-contamination between sampling locations and the transfer of media constituents.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that approved decontamination procedures for all chemical sampling and field analytical equipment are detailed in the project-specific Sample and Analysis Plan prior to the actual field effort and that field personnel required to successfully accomplish the task have been properly briefed and trained.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that sample equipment decontamination procedures utilized during field sampling activities are completed in compliance with this procedure and as detailed in the project-specific Sample and Analysis Plan.

4.3 Field Sampling Team

All members of the Field Sampling Team are responsible for implementing appropriate decontamination procedures as detailed in this procedure and the project-specific Sample and Analysis Plan. Such duties may be performed by geotechnical engineers, field technicians, or other qualified field personnel. Decontamination procedures will be documented in the Field Logbook.

5.0 EQUIPMENT

The following equipment is necessary for the decontamination of sampling equipment:

- Disposable Nitrile Gloves
- Laboratory-Grade, Non-Phosphate Detergent
- Distilled Water
- Contractor-grade plastic trash bags
- Scrub Brushes
- Multi-stage decontamination station
- Five-gallon clean plastic buckets (minimum of 3)
- Polyethylene sheeting

6.0 PROCEDURE

Prior to the collection of samples, equipment used to collect water, soil, sediment, and other samples which makes contact with the sample media will be decontaminated by one of the following methods. Prior to proceeding with decontamination procedures, a decontamination area will be constructed. The minimum requirements for a decontamination area include covering sufficient ground surface area with polyethylene sheeting in order to comfortably conduct all decontamination procedures and minimize conditions resulting in potential cross-contamination. For Method 2 a three-stage/vessel decontamination station should be constructed to progress through the three stages of decontamination. Typically this can consist of clean 5 gallon buckets or other open top vessels or short sections of clean PVC pipe that have been capped on the bottom to retain the decontamination liquids.

Method 1 -- General decontamination procedure for all sampling equipment:

1. Wash and scrub with laboratory-grade, non-phosphate detergent all accessible equipment surfaces. This includes all internal surfaces that are readily accessible and may come into contact with the sample media
2. Rinse with approved distilled water.
3. Rinse a second time with approved distilled water.
4. Wrap in new, unused contractor-grade plastic trash bag, or polyethylene sheeting.

Method 2 -- Decontamination procedure for submersible pumps used to collect groundwater samples for volatile organic compounds:

1. Wash and scrub all accessible surfaces of submersible pumps with laboratory-grade, non-phosphate detergent diluted with distilled water. This includes all internal surfaces that are readily accessible and may come into contact with the sample media.
2. Rinse with distilled water.
3. Immerse the submersible pump in a container filled with laboratory-grade, non-phosphate detergent diluted with distilled water and energize the pump in order to allow the solution to rinse the inaccessible internal lines and chambers of the pump. Detergent to distilled water ration should be at a minimum of 6 grams/gallon.
4. Submerge the pump, in detergent-free distilled water and re-energize the pump in order to rinse the detergent solution from the inaccessible internal lines and chambers of the pump

5. Repeat Step 4 in additional container of detergent-free distilled water.
6. Rinse the pump with deionized water.
7. Wrap in new, unused contractor-grade plastic trash bag, or polyethylene sheeting.

Method 3 -- Decontamination procedure for equipment used to collect metal samples only:

1. Rinse all accessible surfaces of the submersible pump with distilled water.
2. Rinse plastic or Teflon®-coated equipment with 10% nitric acid; rinse stainless steel equipment with 1% hydrochloric acid.
3. Rinse all accessible surfaces, plastic or Teflon®-coated equipment, and stainless steel equipment with deionized, analyte-free water.
4. Air dry.

Bailers and Bailing Line

An elevated potential for cross-contamination between groundwater sampling points via the use of a common bailer, or its attached line, exists unless strict procedures for decontamination are followed. It is recommended that dedicated disposable bailer and bailing line be used for each sample point. Braided nylon or polypropylene lines may be used with a bailer and discarded after each use. If a non-disposable bailer is used it must be decontaminated using procedures outlined for sampling equipment (*Method 1*) prior to the initial groundwater sample collection and to each succeeding sample collection. After completing sample collection at all sample points, the non-disposable bailer should be decontaminated a final time using procedures outlined in *Method 1*.

Sampling Pumps

Most common sampling pumps are low volume pumps (less than two gallons per minute). These include various types of positive displacement pumps. Pumps that allow air or other gases to contact the groundwater sample will not be used. If pumps are used for collecting groundwater samples at more than one sampling point, the pumps will be decontaminated between each sample location. General procedures to be used for decontamination of sampling pumps are described in *Method 2*.

Water Level Indicators

Water level indicators that consist of a probe which contacts groundwater must be decontaminated using the following method:

- Wash and scrub all accessible surfaces of the probe with laboratory-grade, non-phosphate detergent.
- Wash and scrub the measuring tape portion of the meter with laboratory-grade, non-phosphate detergent.
- Rinse the probe and measuring tape portion of the meter with deionized, volatile-free water.
- Wrap the probe in new unused contractor-grade plastic trash bag.

Probes
Probes (e.g., pH or specific ion electrodes, geophysical probes, or thermometers) that come in direct contact with the sample will be decontaminated using the procedure described for Water Level Indicators.

Quality Control Procedures for Decontamination
The effectiveness of field cleaning procedures may be monitored by following quality assurance/quality control procedures (QA/QC) outlined in the project-specific Sampling and Analysis Plan. QA/QC procedures generally consist of collecting periodic equipment blank samples and requesting laboratory analysis for the constituents of concern

Decontamination Notes:
Care will be taken when choosing the location to decontaminate sampling equipment in order to avoid contact with fugitive dust, fuel, oils, gasoline, organic solvents, or any potential source of contamination. All efforts will be made to conduct the decontamination at or adjacent to the sampling location.

Disposal of all investigative derived wastes generated during decontamination procedures is detailed in ERT SOP-06-4 and deviations from the procedure will be described in the project-specific Sample and Analysis Plan.

7.0 REFERENCES

The following documents were referenced in the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Environmental Protection Agency, December 1987. *A Compendium of Superfund Field Operations Methods*, EPA 540/P-87/001.

USEPA, November 1992. *RCRA Ground-Water Monitoring:: Draft Technical Guidance*

United States Environmental Protection Agency, November 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA SW-846, Third Edition.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

Negative Contamination - Occurs when the measured concentration of the analyte is artificially low as a result of volatilization, adsorption, and related losses.

Positive Contamination - Occurs when the measured concentration of the analyte is artificially high due to leaching or the introduction of foreign matter into the sample.

Cross Contamination - A type of positive contamination caused by the introduction of part of one sample with a second sample during sampling or storage.

Detergent - Standard brand of non-phosphate, laboratory-grade detergent such as Alconox or Liquinox.

Acid Solution - A combination of reagent-grade acid and deionized water.

Solvent - Pesticide-grade solvent.

Tap or Potable Water - Water from an approved municipal water treatment system.

Deionized Water - Volatile-free water produced by distillation and procured from an outside party.

Earth Resources Technology, Inc. Standard Operating Procedure **DECONTAMINATION OF DRILLING AND DIRECT-PUSH EQUIPMENT AND MATERIALS**			
Effective Date: **01/29/09**	Version: **001**	ERT SOP-03-2	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/30/09 Date	 Signature	1/29/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide reference information on the proper decontamination of drilling and direct-push equipment, and other materials used in conducting subsurface investigations.

2.0 SCOPE

This procedure addresses decontamination of drilling and direct-push equipment and associated materials only. Personal decontamination guidelines are present in the project-specific work plan. Decontamination of sampling equipment is described in ERT SOP-03-1.

3.0 REQUIREMENTS

To ensure that chemical analysis results are reflective of the actual concentrations present at sampling locations, various drilling and direct-push equipment used in subsurface investigations must be properly cleaned and decontaminated minimizing the potential for cross-contamination and transfer of constituents between subsurface sample locations.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that approved decontamination procedures for all drilling and direct-push equipment used for subsurface investigation are detailed in the project-specific Sample and Analysis Plan prior to the actual field effort and that field personnel required to successfully accomplish the task have been properly briefed and trained.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that equipment decontamination procedures utilized during field sampling activities are completed in compliance with this procedure and as detailed in the project-specific Sample and Analysis Plan.

4.3 Field Team

The Field Team is responsible for performing decontamination procedures in accordance with this procedure and the project-specific work plans. Such duties may be performed by geotechnical engineers, field technicians, or other qualified field personnel. Decontamination procedures will be documented in the Field Logbook.

5.0 EQUIPMENT

- Portable Hot Water Pressure Washer
- Portable water supply tank with necessary hose connections
- Insulated Gloves

- Rubber Boots
- Safety glasses
- Potable water
- 6 mil or greater Plastic Sheeting
- Berm Construction Materials
- Liquid Transfer Pump
- 55-Gallon Drums

6.0 PROCEDURE

Pressure Washing
All drilling and direct-push equipment involved in subsurface investigative activities will be decontaminated by heated pressure washing prior to drilling activities at each borehole. Pressure washing will be performed over a designated decontamination area, as described below.

Standard Washing Protocol
All down-hole tooling and materials involved in subsurface investigative activities that come into direct or indirect contact with subsurface material will be decontaminated by a standard washing protocol. Such equipment and materials include augers, drive rods, and drill bits. Decontamination of tooling used to collect samples, such as split spoons and macro cores, will be conducted as per ERT SOP-03-1.

The following general decontamination method will be utilized for drilling and direct-push equipment:

- Place equipment to be washed on an elevated platform inside of the designated decontamination area
- Use a brush or broom to remove heavy soil spoils
- Pressure wash all accessible surfaces using clean potable water
- Allow to air dry
- Handle and transport to next boring location using care to retain the cleanliness of the equipment. When practical transport on new unused polyethylene plastic sheeting.

Pressure washing will be performed over a designated decontamination area, as described below.

Screen and casing (PVC) supplied by the manufacturer that is free of manufacturing markings, residues and labeling, and sealed in plastic will not require decontamination.

Designated Decontamination Areas
A designated decontamination area will be established to contain decontamination wastes and waste waters. The design of the decontamination area may vary but at a minimum must be of adequate size and structural composition to contain decontamination waste. This may consist of a portable damn or self constructed berm, lined with minimum 6-mil plastic. The berm and liner will be assembled in such a way as to contain all liquid IDW generated by the decontamination procedure. The location of the decontamination area will be identified in the project-specific work plan and subject to site conditions.

Liquid IDW will be collected and containerized (i.e. 55-gallon DOT drums, plastic water tote) for eventual disposal at an approved facility in accordance with the project-specific work plan.

7.0 REFERENCES

The following documents were referenced in the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Environmental Protection Agency, November 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* EPA SW-846, Third Edition.

United States Environmental Protection Agency (USEPA), 1991. *Handbook of Suggested Practices for the Design and Installation of Ground Water Monitoring Wells*. EPA160014-891034. March.

United States Environmental Protection Agency (USEPA), 1992. *RCRA Ground Water Monitoring: Draft Technical Guidance.* November

United States Army Corp of Engineers (USACE), 1994. *Monitor Well Design, Installation, and Documentation at Hazardous and/or Toxic Waste Sites*. EM 1110-1-4000. November.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

Cross Contamination - Type of positive contamination caused by the introduction of part of one sample with a second sample during sampling or storage**.**

Detergent - Laboratory-grade non-phosphate detergent such as Alconox or Liquinox.

Tap or Potable Water - Water from an approved municipal water treatment system.

IDW – Investigative Derived Waste

Earth Resources Technology, Inc. Standard Operating Procedure **MONITORING WELL INSTALLATION**			
Effective Date: **01/29/09**	Version: **001**	**ERT SOP-05-2**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/29/09 Date	 Signature	1/28/09 Date

1.0 PURPOSE

The purpose of this procedure is to establish generally acceptable methods for proper monitoring well design and construction.

2.0 SCOPE

This procedure is applicable to the construction of semi-permanent monitoring wells at field investigation sites. The methods described herein include multiple options to account for the variety of conditions that may be encountered in the field. Specific monitoring well installation procedures proposed for use are described in the project-specific work plan.

3.0 REQUIREMENTS

The objectives for each monitoring well and its intended use will be clearly defined before the monitoring well is constructed. Within a monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well construction, attention must be given to clearly documenting the basis for construction decisions, as-built details of well construction, and the construction materials used.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for making final decisions regarding monitoring well construction details and ensuring contract obligations are fulfilled.

4.2 Field Team Leader

The Field team Leader is responsible for ensuring that field personnel have been trained in the use of this procedure. The Field Team Leader is responsible for overseeing the installation of monitoring wells ensuring accurate construction, ensuing as built construction logs are created for each installed well and ensuring site safety protocols are followed to during field activities.

4.3 Field Team

The Field Geologist is responsible for ensuring the well is installed according to the contract specifications and documenting as-built well construction details.

5.0 EQUIPMENT

- Field Logbook and Indelible Ink Pens
- Monitoring Well Construction Log

- Soil Boring Log
- Folding or Retractable Engineers Rule (calibrated to 0.01 foot)
- Weighted Tape (calibrated to 0.01 foot)
- Slot Size or Feeler Gauge
- Water-Level Indicator
- Copies of the Statement of Work, Sampling and Analysis Plan/Field Sampling Plan, Site Safety and Health Plan, and approved Work Plan
- 10X Hand Lens

6.0 PROCEDURE

The following procedures are to be followed by the well installation contractor. The ERT Team Leader will be responsible for providing oversight to the well installation contractor, ensuring the procedures below are followed, and documenting the well construction details.

6.1 Design Considerations

6.1.1 Monitoring Well Depth, Diameter, and Screen Length

Standard monitoring well diameters are two, four, six, or eight inches. For most monitoring programs, a two- or four-inch diameter monitoring well is preferred. Smaller wells have a smaller volume of stagnant water, well construction costs are lower, and the water table stabilizes more readily.

- The stagnant water in a monitoring well available for sampling after complete recharge is dependent on the well diameter as follows:

Casing Inside Diameter (inches)	Standing Water Depth to Obtain One Gallon Water (ft)	Total Depth of Standing Water For Four Gallons (ft)
2	6.13	25
4	1.53	6
6	0.68	3

- The borehole diameter used to insert the monitoring well should be at least four inches larger than the well riser pipe diameter to account for adequate annular spacing around the well casing.
- Wells constructed in a 100-year flood plain must be flood protected by either sealing the well or extending the well casing above the established 100-year flood elevation.
- Monitoring wells will contain only one screened interval per well unless specified in project-specific work plans. Separate boreholes will be drilled for cluster monitoring wells whose purpose is to monitor several intervals.

6.1.2 Riser Pipe and Screen Materials

Schedule 40 PVC has sufficient tensile and compressive strength for wells up to 1200 feet and is considered generally acceptable. Project specific modifications to monitoring well construction will be details in the project Sample and Analysis Plan. All well construction materials must be decontaminated before using according to ERT SOP-03-2.

- Type 316L stainless steel or other alloys will be considered for use in sulfidic waters.
- Threaded, flush joint casing is required. No glues, solvents or rubber O-rings will be used in well construction.
- For deep wells, the screen must be chosen to withstand the column weight without collapsing. The screen shall pass no more than 10 percent of pack material on in-situ aquifer material.
- The well screens to be used will have a slot size of 0.010 inches, and the gravel pack mesh size will be 10/20, unless deemed improper by the field geologist.
- Materials other than stainless steel, PVC, and Polytetrafluoroethylene (PTFE) will not be used in the construction of monitoring wells.
- Screen lengths will generally be 10 feet long, except where the aquifer to be examined is less than 10 feet thick, where a particular contaminant layer is being screened, or specified by the statement of work. A five-foot screen length or less will be installed in these instances. Well screens shall be placed no more than three feet above the bottom of the drilled borehole.

6.1.3 Annular Materials

Material placed in the annular space between the borehole and well riser pipe includes a granular filter pack, when necessary, a bentonite seal, and/or cement grout. Generally, all of these materials should be installed via a rigid tremie pipe (unless alternate methods are specified) placed in the annular space with the exception of bentonite pellets. The granular filter pack is usually a medium- to very coarse-grained uniform sand. The quantity of sand placed in the annual space is dependent upon the length of the screened interval, borehole diameter, and casing size. The granular filter pack should extend a minimum of two feet above the top of the screened interval and a maximum of 5 feet for well depths greater than 200. Granular filter packs can be poured (free fall) into the annular space of the borehole/auger when wells are relatively shallow (less than 30 feet in total depth)

A bentonite pellet seal will be installed atop the granular filter pack and be a minimum of two feet thick and a maximum of five feet thick. A bentonite slurry seal will be used only as a last resort because betonite slurries have a thick consistency and are more difficult to homogenize with the annular space. Bentonite pellets can be placed above the filter pack by dropping them directly down the annulus in shallow wells (less than 30 feet in total depth) or in wells slightly deeper where the water table is close to the screened interval and the borehole is competent. Otherwise they will be introduced via a tremeie pipe as previously detailed. Bentonite expands by absorbing water and provides a seal between the screened interval and the rest of the annular space and formation.

Cement grout is then placed in the annular space from the top of the bentonite seal to the ground surface. The seal, whether all bentonite or bentonite capped with grout, prevents surface runoff from reaching the screened interval. Backfilling the annular filling with bentonite and grout also prevents borehole collapse and helps ensures integrity of the well construction. A side discharge, rigid tremie pipe will be used to introduce bentonite slurry and grout from the bottom of the borehole upward to prevent bridging and to provide a better seal.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation process, grout refers to solidified material which is installed and occupies the annular space above the bentonite seal. Grout generally is composed of two assemblages of material (i.e., a cement-bentonite grout or a neat cement grout). A cement-bentonite grout normally is a mixture of cement, bentonite, and water, up to five pounds granular or flake-type bentonite and up to eight gallons of water per 94-pound bag of Portland Type II or V cement. Neat cement is made up of one 94-pound bag of Portland Type II or V cement and up to eight gallons of water.

6.1.4 Protective Equipment

A protective steel casing will be installed around any portion of the well which extends above the ground surface. . The protective casing will be installed at the same time grout is poured in the annular space around the well. The casing will have a hinged cap and can be locked to prevent vandalism. A slide-on cap that fits snuggly on the PVC well riser will be utilized to prevent unwanted material from entering the well and maintaining static atmospheric pressure as water levels rise or fall with the exception of wells installed to monitor gases. The top of the protective casing to the top of the well casing will be no more than 0.5 feet. The protective casing will have a larger diameter than the well and be set in the grout.

Four 3-inch diameter steel guard posts filled with cement may be placed around the protective steel riser pipe. Guard posts are generally six feet in total length and installed approximately three feet into the ground with independent concrete footings. A concrete pad will be placed around the installed protective casing, unless otherwise specified in the statement of work. Fencing also will be installed, if applicable. In addition, at least one ¼-inch hole is drilled just above the concrete pad through the protective casing which acts as a drainage port for the flow of water which may enter the annulus during well development, purging, or sampling.

When appropriate, wells may be finished with a flush mount. With this method, the protective casing is cut at the ground surface and the top of the well is cut approximately six inches below the ground surface. A protective sleeve is set into the wet cement around the well with the top set level with the ground surface. A locking gasket cap is generally placed on the well to seal out water and a manhole type lid placed over the protective sleeve. The top of the well resembles a small manhole, typically less than 8 inches in diameter. If the cement grout seal is effective and does not leak, the hole below the pavement will hold water. A drainage system or periodic purging may be required to direct pooled water away from the well head.

6.2 Monitoring Well Construction

6.2.1 Pre-drilling Activities

Available underground utility maps for the immediate vicinity of a drilling site will be reviewed and proposed drilling locations will be staked in the field for inspection. One Call or Miss Utility will be contacted and provided adequate time to mark all utilities in the immediate vicinity of activities. Digging permits will be obtained. Utility mark outs and obtaining permits must be accomplished before performing drilling activities.

6.2.2 Well Construction (see Attachments 1-3)

- Prior to installation, inspect the screen to insure that no damage has occurred during shipment and decontamination. Record the type of construction material, the class of material and screen slot size. If necessary, check the slot size with a feeler gauge to insure that the screens are properly labeled.
- Prior to placing well materials in the borehole, place a backfill of filter pack material below the base of the screen not to exceed three feet. The filter pack will consist of chemically inert (e.g., clean quartz sand, or silica) material and of appropriate size for the well screen and host environment.
- Assemble the well casing and screen, and place the materials in the borehole. Ensure that each well is as straight as possible and centered in the borehole. All screen bottoms shall be securely fitted with a threaded cap or plug with a maximum length of 0.5 feet. Stabilizers will be used for wells greater than 150 feet.
- Record the depth of the base of the well, the top of the screen, and the screen length in the Monitoring Well Construction Log (see Attachment 4).

NOTE: All well screen, riser pipe, bottom plug, and cap will be threaded and flush jointed. No glues, solvents or rubber O-rings are to be used in monitoring well installation.

6.2.3 Placement of Filter Pack and Annular Seal

- Place the monitoring well filter pack into the annulus between the well screen and the open borehole wall by slowly pouring filter pack material directly or pumping sand slurry through a rigid tremie pipe. If the borehole will not stand open, place filter pack material directly into the annulus between the auger wall and the well casing and screen. Then gradually pull back the auger line in short increments (two feet at a time) to allow the sand to settle around the screen below the augers. Care will be taken to prevent filter pack material from bridging between the borehole wall or augers and the well screen and riser pipe. A weighted tape will be used periodically to verify the placement of the filter pack.
- The filter pack material will be placed from the bottom of the well to between two and five feet above the top of the screen. The depth to the top of the sand pack will be recorded on the Well Construction Log. The amount (by weight) and volume of sand used will also be noted.
- For shallow wells (<35 feet), gravity feed bentonite pellets (not powder) onto the top of the filter pack to form the bentonite seal. Bentonite pellets are generally used and preferred. If a tremie pipe is used, slowly withdraw the pipe as the bentonite is added to ensure even placement around the annulus. Periodically check the depth and consistency with a weighted tape. Pellet seals shall be a minimum of two feet thick as measured immediately after placement, without allowance for swelling.
- Follow manufacturer's specifications for hydration procedures. Record the depth to the top of the bentonite seal, the amount of bentonite used, the amount of water added for hydration, if applicable, and compare actual volume used with calculated volume on the Monitoring Well Construction Log (see Attachment 4).

NOTE: The annular seal material must be chemically compatible with the well materials and contaminants and chemically inert so it does not affect the quality of groundwater samples. The permeability of annular seals will be one to two orders of magnitude less than the surrounding formation. Filter pack material will be chemically and texturally inert (e.g., clean quartz sand, or silica) with particles that are of appropriate size.

Tremie cement-bentonite grout into the annulus above the bentonite seal by pumping it through a side-discharge, rigid tremie pipe, until reaching the ground surface. If the bentonite seal is less than 10 feet below the ground surface, the grout may be poured into the annulus. If drill casing is still in the borehole, it should be pulled at this time, and more grout added to compensate for settling. Place the steel protective well casing into the fresh grout. After 24 hours, add more grout, if settling has occurred. This process will be repeated until firm grout remains at the ground surface.

NOTE: Mix the cement and water first, and then add the bentonite. All prescribed portions of grout shall be combined in an above-ground, ERT-approved container and mechanically (not manually) blended to produce a thick, lump-free mixture throughout the mixing vessel.

6.2.4 Above Ground Well Completion

- Make a V-notch on the north side of the well casing with a file or knife. The notch will be the point from which surveys and subsequent groundwater level measurements will be taken. Ensure no filings or shavings enter the well.
- For wells that are completed above the ground surface, the steel protective casing should extend approximately 2.5 feet above the ground surface. The protective casing should be equipped with a hinged, loose-fitting cap that can be locked to prevent unauthorized entry. Sufficient space must be allowed between the protective casing lid and the top of the well riser pipe for a well cap. The minimum size for a two-inch diameter well is four-inch diameter protective casing, for a three-inch diameter well is five-inch diameter protective casing, for a four-inch diameter well is six-inch diameter protective casing, and for a five-inch diameter well is eight-inch diameter protective casing. The top of the protective casing to the top of the riser pipe shall be no more than 0.5 feet.
- Construct concrete pad around the protective casing after the grout has finished settling. The pad shall slope away from the casing in all directions.
- Drill a ¼-inch diameter drainage port into the side of the protective casing approximately 1/8 inches above the internal mortar collar to permit drainage.
- In some instances, install four, 3-inch diameter by 6-foot long, concrete filled steel guard posts equidistant around the concrete pad. The guard posts will extend approximately three feet into the ground with independent concrete footings.

6.2.5 Flush Mount Completions

For wells that are completed flush with the ground surface, a well vault will be installed over the well riser pipe. The vault will be water-tight and equipped with a locking mechanism to prevent unauthorized entry. For flush-mount completions, the top of the well is 4- to 5-inches below ground surface and concreted to at least six inches below ground surface.

6.2.6 Monitoring Well Installations in Confined Aquifers

1. Advance an oversized borehole through unconsolidated surface deposits to a depth of 2- to 3-feet below the top of the confining bed. In general, the borehole should be 2 inches in diameter larger than the casing to be installed when a tremie pipe is to be used.
2. Condition the borehole until the hole is cleared of cuttings.
3. While performing Step 2, construct the necessary length(s) of surface casing. Surface casing may be of mild or galvanized steel.
4. Pressure grout bentonite pellets or chips to fill the portion of the borehole in the confining bed. If the bentonite seal is to be set below the static water level, only pellets may be used. The bentonite should hydrate in the presence of groundwater, but approved potable or distilled water may be added, if needed.
5. Insert the surface casing into the borehole and set firmly into the bentonite seal in the confining bed.
6. Mix Portland cement and water to make a pumpable slurry.
7. Insert the tremie pipe into the borehole and begin pumping grout. Slowly withdraw the tremie pipe as the annulus fills to ensure even placement with no bridging
8. Allow grout to cure for 48 hours or longer before proceeding.
9. After grout has cured, advance with a smaller diameter bit and proceed drilling. Advance the borehole to the desired depth. The hole should be drilled a few feet deeper than necessary to allow for cave-ins during casing placement. If more than one aquifer will be encountered during drilling, the well must be cased in separate states to prevent cross-contamination. Steps 1 through 8 of this section will be followed for each separate aquifer that must be cased off.
10. Condition the borehole until the hole is cleaned of cuttings. Pull the drill string out of the borehole when no additional cuttings reach the surface. Check the total borehole depth with a weighted surveyor's tape.
11. Construct the casing string in manageable sections while conditioning the borehole. The casing and screen (if used) must be decontaminated in accordance with ERT SOP-03-2 before make-up. Tighten casing joints to the manufacturer's specifications.
12. Insert the first segment of the casing string and lower to a manageable height for adding the second casing segment. Chock the casing, add the second segment, then release the chock and lower the casing. Repeat this process until the full casing string is suspended in the well. Centralizers should not be placed anywhere on the well riser or screen that will be covered with the filter pack. The centralizers should be placed at 25 foot intervals. The casing string should be allowed to hang in the well rather than set on the bottom. Casing strings with Teflon® screens should never be set on the bottom because the weight of casing will significantly reduce the slot size and may collapse the screen.
13. Insert the tremie pipe so it is near the bottom of the screen and begin placing the filter material into the borehole. Slowly withdraw the tremie pipe so that the filter pack is placed evenly around the screen without bridging. A maximum of three feet of filter pack material may be spotted at the bottom of the hole, under the screen. The filter pack must be installed to five feet above the top of each screened interval. If the top of the screen is below the bottom of the confining layer, extend the filter pack to the confining

layer, as appropriate. Develop the well according to ERT SOP-05-4 to settle the filter pack and remove slurry water.

14. If the filter pack was placed as slurry, withdraw the tremie pipe, rinse with potable water, and dry before proceeding to add the bentonite seal. The potable rinse water does not have to be containerized. If the filter pack was installed dry, do not remove the tremie unless a larger diameter pipe is needed for installing the bentonite seal. Check the depth of the filter pack to ensure that it adequately rises above the top of the screen.
15. Pour bentonite pellets onto the top of the filter pack. Bentonite pellets must be used for installations below the water table. The bentonite seal should extend a minimum of two feet into the confining layer, if possible. Slowly withdraw the tremie pipe as bentonite is added to ensure even placement around the casing and prevent bridging.
16. Hydrate the bentonite according to the manufacturer's specifications. Record amount of water used.
17. Mix Portland cement with powdered bentonite (as previously described) and water to make a pumpable slurry.
18. Tremie the grout into the annulus using a side-discharge tremie pipe. Slowly withdraw the tremie pipe as the annulus fills to ensure even placement. Grout the well to the ground surface.
19. After installing grout, dismantle and clean the tremie equipment.

6.2.7 Monitoring Well Completion and Borehole Records

The Field Geologist will complete a Soil Boring Log and Well Installation Log (see Attachment 5) for all borings within 10 working days after well installation. Borehole logging will be in accordance with ERT SOP-07-3.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

American Society of Testing and Materials, 1989. *Proposed Recommended Practice for Design and Installation of Groundwater Monitoring Wells in Aquifers*. ASTM Subcommittee D18-2105 on Design and Installation of Groundwater Monitoring Wells.

Driscoll, Fletcher, G. 1986. *Groundwater and Wells*, 2nd Edition, Johnston Division, St. Paul, Minnesota, pp. 1089.

HAZWRAP, July 1990. Quality Control Requirements for Field Methods, DOE/HWP-69/R1.

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 1994. *Monitor Well Design, Installation, and Documentation at Hazardous and/or Toxic Waste Sites*. EM 1110-1-4000. November.

USEPA, November 1992. RCRA Ground-Water Monitoring: Draft Technical Guidance.

United States Environmental Protection Agency, September 1986. Resource Conservation and Recovery Act (RCRA) Ground Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

United States Environmental Protection Agency (USEPA), 1991. *Handbook of Suggested Practices for the Design and Installation of Ground Water Monitoring Well*. EPA160014-891034. March.

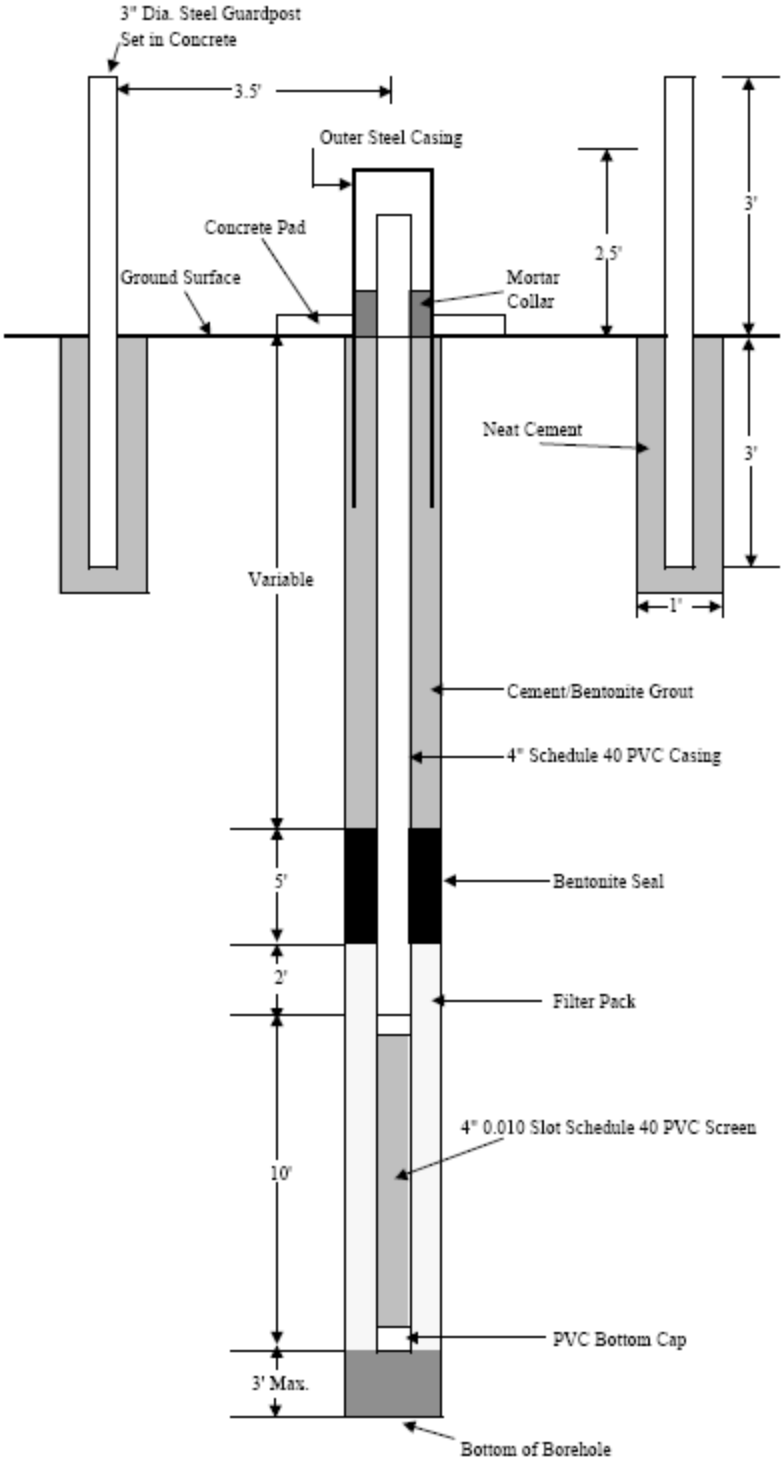
Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology,* McGraw-Hill, New York

8.0 DEFINITIONS

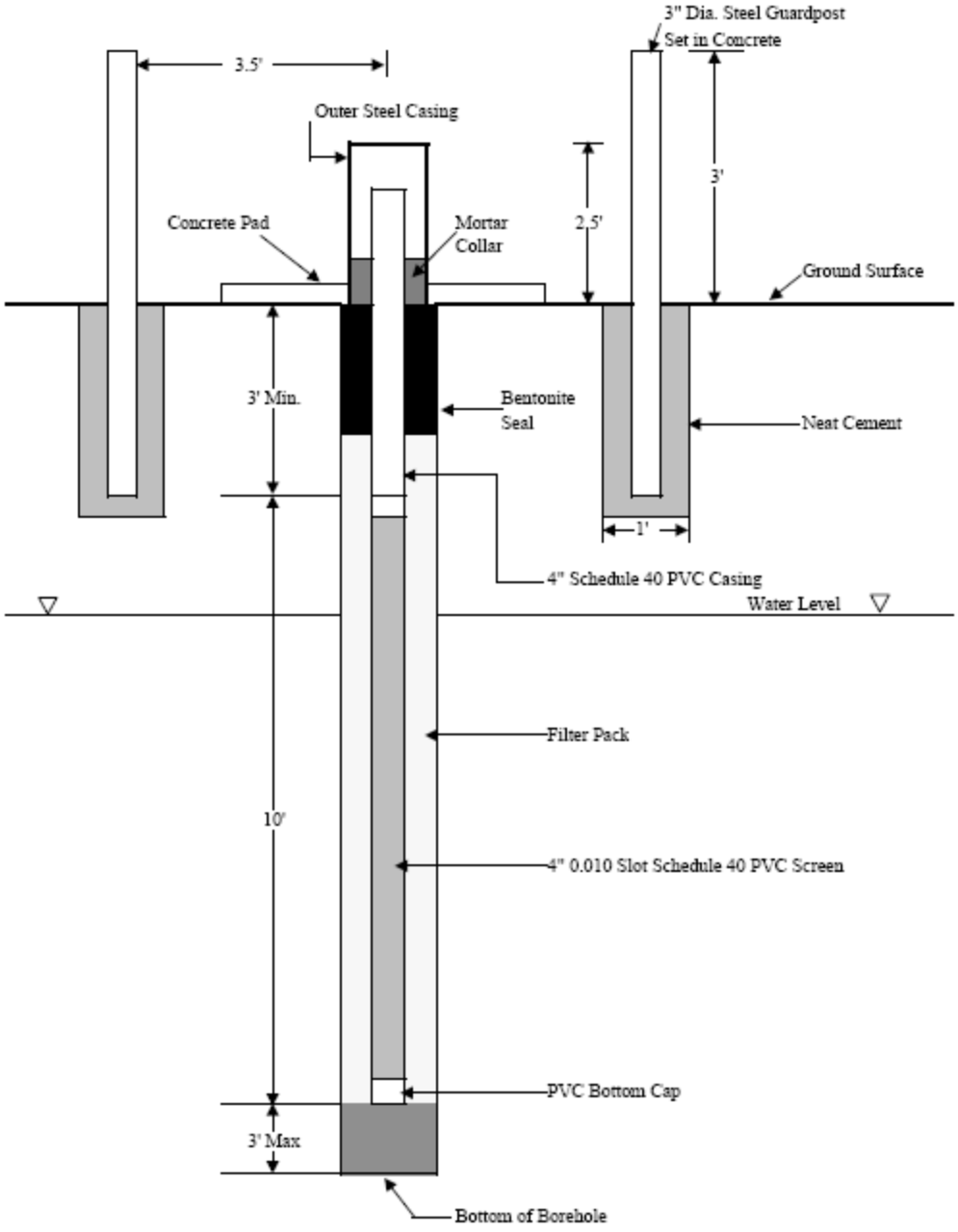
Monitoring Well - A well which will provide for the measurement of total well depth, the collection of representative groundwater samples, the detection and collection of representative light- and dense-phase organics, and measurement of water levels and water flows.

9.0 Attachments

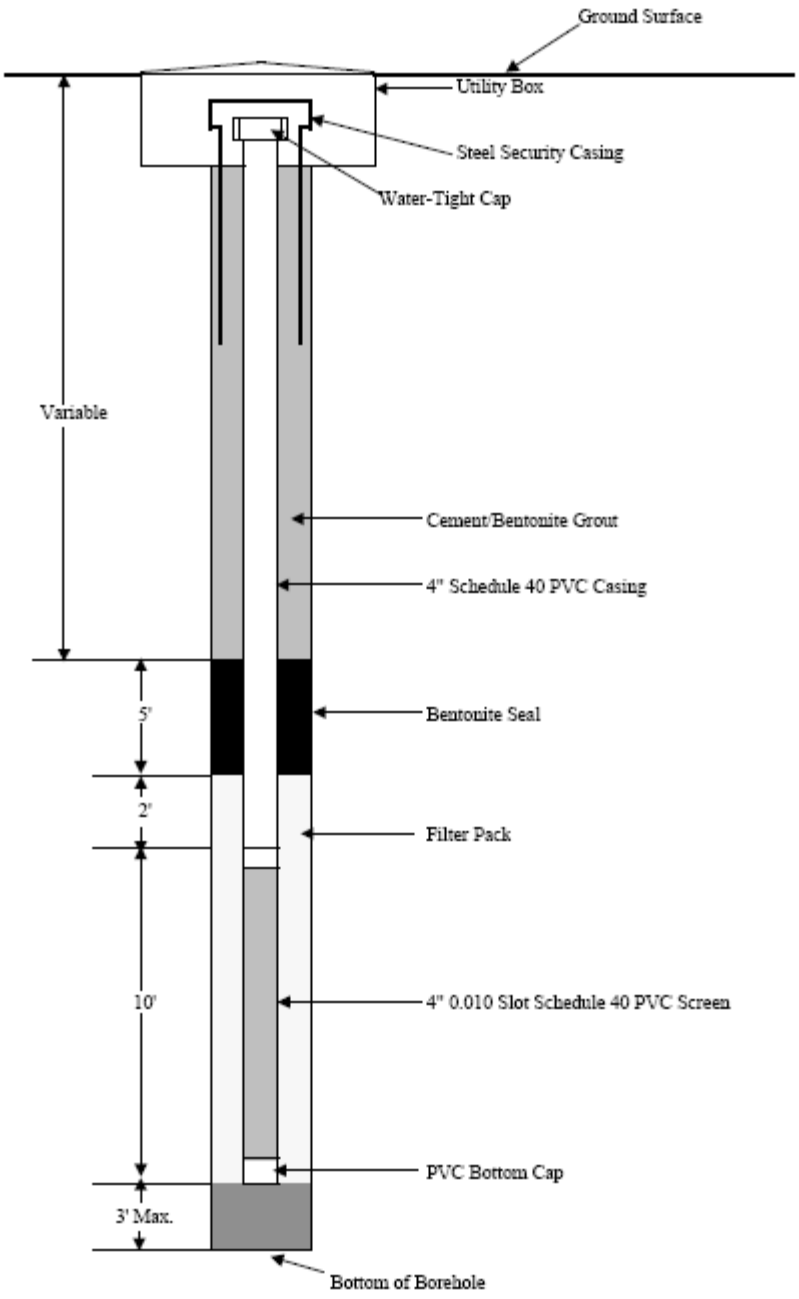
ATTACHMENT 1
Standard Monitoring Well Construction



ATTACHMENT 2
Monitoring Well Construction When Water Table is Near Land Surface



ATTACHMENT 3
Monitoring Well Construction With Sealed Cap and Flush Surface Presentation



ATTACHMENT 4
Monitoring Well Construction Log

Earth Resources Technology

WELL CONSTRUCTION DIAGRAM

	PROJECT NAME	WELL NUMBER
	PERMIT NUMBER	DATE INSTALLED (START)
	WELL DISTANCE FROM SOURCE	DATE INSTALLED (COMPLETED)
TYPE OF PROTECTIVE COVER STICK UP ☐ FLUSH MOUNT ☐	LOCATION OF WELL RELATIVE TO SOURCE ☐ UP GRADIENT ☐ SIDE GRADIENT ☐ DOWN GRADIENT ☐ UNKNOWN	WELL INSTALLED BY

USCS CLASSIFICATION OF SOIL NEAR SCREEN

GP ☐ GM ☐ GC ☐ GW ☐

SW ☐ SP ☐ SM ☐ SC ☐

ML ☐ MH ☐ CL ☐ CH ☐

DRILLING METHOD USED
ROTARY ☐
HOLLOW STEM ☐
OTHER ☐

DRILLING FLUID USED
WATER ☐ AIR ☐
MUD ☐ NONE ☐

RISER INTERVAL _____ TO _____ ft BGS

SCREEN INTERVAL _____ TO _____ ft BGS

ANNULAR SPACE SEAL _____ TO _____ ft BGS

BENTONITE SEAL _____ TO _____ ft BGS

SECONDARY FILTER _____ TO _____ ft BGS

PRIMARY FILTER _____ TO _____ ft BGS

WELL BOTTOM _____ ft BGS

FILTER PACK BOTTOM _____ ft BGS

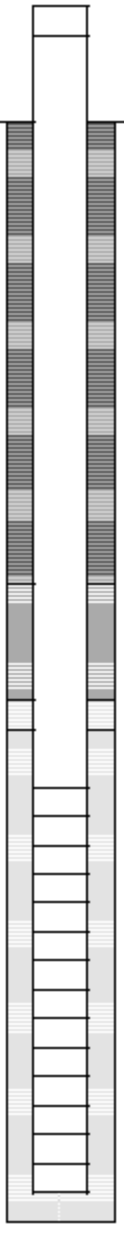
BOREHOLE BOTTOM _____ ft BGS

BOREHOLE DIAMETER _____ inches

ID WELL CASING _____ inches

WATER LEVEL AFTER COMPLETION _____ ft TOC

NOTES:



1 CAP AND LOCK YES ☐ NO ☐
2 PROTECTIVE POSTS YES ☐ NO ☐
3 PROTECTIVE CASING YES ☐ NO ☐
INSIDE DIA _____ in
LENGTH _____ ft

4 ANNULAR SPACE SEAL
_____ lb/gal GRANULAR BENTONITE ☐
_____ lb/gal BENTONITE SAND SLURRY ☐
_____ lb/gal BENTONITE SLURRY ☐
BENTONITE - CEMENT GROUT ☐
% BENTONITE
HOW INSTALLED
TREMIE ☐
TREMIE PUMP ☐
GRAVITY ☐

5 CENTRALIZERS ☐ YES ☐ NO

6 BENTONITE SEAL
GRANULES ☐
PELLETS ☐
OTHER ☐
_____ BAGS/SIZE

7 SECONDARY FILTER ☐ YES ☐ NO
BAGS/SIZE _____

8 FILTER PACK MATERIAL NAME/SIZE
_____
_____ BAGS/SIZE

9 WELL CASING
PVC SCHD 40 ☐
PVC SCHD 80 ☐
_____ OTHER ☐

10 SCREEN MATERIAL:
TYPE: _____ FACTORY CUT ☐
CONT. SLOT ☐
MANUFACTURER _____
SLOT SIZE _____ INCHES
SLOTTED LENGTH _____ FT

11 BACKFILL MATERIAL: (BELOW FILTER PK)
NONE ☐
OTHER ☐

ATTACHMENT 5
Soil Boring Log

Earth Resources Technology

BORING LOG: SHEET OF

PROJECT NAME:	PROJECT NUMBER: DATE:
LOCATION:	ADDRESS:
DRILLING CONTRACTOR:	DRILLER
DRILL RIG TYPE:	DRILLING METHOD:
SITE SKETCH	GEOLOGIST:
	SAMPLE METHOD:
	BORING FT DIA IN
	WELL DEPTH FT
	WELL DIAMETER IN
	RISER TO
	SCREEN TO
	PERMIT NUMBER:

DEPTH	Geologic Description	USCS	FIELD SCREENING RESULTS	BLOW COUNT	RECOVERY	SAMPLE ID	INTERVAL

GRANULAR MATERIAL	SIZE DESCRIPTION	SIZE
BOULDERS		> 23 cm (9 in)
COBBLES		7.6 cm (3 in) - 23cm (9 in)
GRAVEL	Coarse	25 mm (1 in) - 76 mm (3 in)
	Medium	9.5 mm (0.375 in) - 25 mm (1 in)
	Fine	2 mm (0.08 in) - 9.5 mm (0.375 in)
Sand	Coarse	0.59 mm - 2 mm
	Medium	0.25 mm - 0.59 mm
	Fine	0.074 mm - 0.25 mm

GRANULAR MATERIAL	SMALLEST DIAMETER OF ROLLED THREADS
Silt	none
Clayey SILT	6 mm (1/4 in)
SILTand CLAY	3mm (1/8 in)
CLAY and SILT	1.5 mm (1/16 in)
Silty Clay	0.8 mm (1/32 in)
CLAY	0.4 mm (1/64 in)

MAJOR DIVISIONS			LETTER SYMBOL	TYPICAL DESCRIPTION
COARSE GRAIN SOILS MORE THAN 50% OF MATERIAL IS LARGER THAN NO.200 SIEVE SIZE	GRAVEL AND GRAVELY SOILS MORE THAN 50% OF COARSE RETAINED IN NO.4 SIEVE	CLEAN GRAVELS LITLE OR NO FINES	GW	WELL GRADED GRADED GRAVELS, GRAVEL SAND MIXTURES, LITTLE OR NO FINES
			GP	POORLY GRADED GRAVELS, GRAVEL SAND MIXTURES, LITTLE OR NO FINES
		GRAVELS WITH FINES APPRECIABLE AMOUNT OF FINES	GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES
			GC	CLAYEY GRAVELS, GRAVEL-SAND-CLAY MIXTURES
	SAND AND SANDY SOILS MORETHAN 50% OF COARSE FRACTION PASSING NO.4 SIEVE	CLEAN SAND LITTLE OR NO FINES	SW	WELL GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES
			SP	POORLY GRADED SANDS, GRAVELLY SAND, LITTLE OR NO FINES
		SANDS WITH FINES APPRECIABLE AMOUNT OF FINES	SM	SILTY-SANDS, SAND-SILT MIXTURES
			SC	CLAYEY SANDS, SAND-CLAY MIXTURES
FINE GRAINED SOILS MORE THAN 50% OF MATERIAL IS SMALLER THAN NO.200 SIEVE SIZE	SILTS AND CLAYS LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS OR CLAYEY SILTS WITH SLIGHT PLASTICITY
			CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
			OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
	SILTS AND CLAYS LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SAND OR SILTY SOILS
			CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS
			OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICTY, ORGANIC SILTS
HIGHLY ORGANIC			PT	PEATS, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENT

Earth Resources Technology, Inc. Standard Operating Procedure **WELL DEVELOPMENT**			
Effective Date: **01/26/09**	Version: **001**	**ERT SOP-05-4**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/26/09 Date	 Signature	1/26/09 Date

1.0 PURPOSE

The purpose of this procedure is to define the requirements for developing monitoring wells with the intent to increase permeability around the well screen and ensure a representative groundwater sample can be obtained from the water-bearing zone.

2.0 SCOPE

This procedure applies to the development of wells by either bailing or pumping techniques. Development water will be staged onsite in appropriate containers, characterized by laboratory analysis, and handled in accordance with ERT SOP-06-4 and the project-specific Sample and Analysis Plan.

3.0 REQUIREMENTS

The purpose of well development is to stabilize and increase the permeability of the filter pack around the well screen, and restore the permeability of the local formation which may have been reduced by drilling and installation operations. The selection of well development methods, well construction and installation details, and the characteristics of the formation in which the well is to be screened, will be detailed in the project-specific Sample and Analysis Plan. Any equipment introduced into the well or in contact with development water will be decontaminated in accordance with ERT SOP-03-1 or ERT SOP-03-02, as applicable.

Typically monitoring wells will be developed by removing a minimum of 3 well volumes and a maximum of 5 well volumes of groundwater from the well. The Field Team Leader will be required to make a visual observation of the resulting groundwater conditions following development and the description will be noted in the Field Logbook. Typical observations include the color, odor, turbidity (non-soluble constituents) and volume of water removed. The purpose for developing a well is not to stabilize groundwater quality indicators, as is typical for environmental sampling and therefore, physical and chemical parameters including temperature, pH, specific conductance and turbidity will not be measured during well development.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that field personnel have been adequately trained and that appropriate well development procedures are detailed in the project-specific Sample and Analysis Plan.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that well development activities are performed in accordance with this procedure and the project-specific Sample and Analysis Plan. At the completion of each well development, the Field Team Leader will inspect the resulting water and record the observation in the Field Logbook, including color, odor, turbidity (non-soluble constituents) and volume of water removed, and other information as detailed in the project-specific Sample and Analysis Plan.

4.3 Field Team

The Field Team is responsible for developing monitoring wells in accordance with this procedure and the project-specific Sample and Analysis Plan. Pertinent information will be recorded in the Field Logbook, including the color, odor, turbidity (non-soluble constituents) and volume of water removed from the well, and other information as detailed in the project-specific Sample and Analysis Plan

5.0 EQUIPMENT

- Pump
- Surge blocks and accessory equipment
- Bailers
- Polypropylene Rope for Bailer Line (or approved equal)
- Water Level Indicator
- Photoinonization Detector
- Drums or Mobile Tanks to Contain the Development Water
- Field Logbook
- Well Development Log

6.0 PROCEDURE

6.1 Development Methods

6.1.1 Mechanical Surging

Operation of a piston-like device termed a surge block affixed to the end of a length of drill rod, or drill stem, is a very effective development method that can be utilized in all diameter of wells, even in stratified formations having variable permeability. The up-and-down plunging action alternately forces water to flow into and out of the well, similar to a piston in a cylinder. The use of a surge block can agitate and mobilize particulates around the well screen. Periods of surging should be alternated with periods of water extraction from the well so that sediment, brought into the well, is removed. Surging should initially be gentle to assure that groundwater can freely enter and exit the well, and that the surge block is not so tight as to damage the well riser or screen. For short well screens (1.6 m (5 ft) or less) set in a homogeneous formation, the surge block does not have to be operated within the screen interval. However, if the screened interval includes constituents of high and low permeability, the block may have to be operated gently within the screened portion of the well.

6.1.2 Pumping

A commonly used development method consists of removing water from the well using a pump set at a high rate. This over-pumping technique, is generally successful in relatively non-stratified, clean-sand formations. By pumping the well at a rate greater than that used during

sampling events, mobilized particulates may be removed, thereby providing a cleaner well for sampling. Over-pumping may be supplemented with the use of a bottom discharge/filling bailer for removing heavy sediment fines. During development, the pump should periodically be raised and lowered in order to agitate deposited sediments in order to extract sediments suspended in the removed water. A disadvantage of development using a pump is that smaller soil grains of the filter pack may bridge the well screen or filter pack, due to one directional flow of water. To correct this potential problem, over-pumping is often used in conjunction with backwashing or surging.

6.1.3 Backwashing

Backwashing is the process of forcing water down and out of the well through the well screen, causing soil particles to dislodge that may have become wedged in or bridged around the well screen due to over-pumping of the well. Backwashing when supplemented with over-pumping, facilitates the removal of fine-grained materials from the formation surrounding the well. A commonly used backwashing procedure called "rawhiding" consists of starting and stopping the submerged pump intermittently in order to allow water in the well pipe to fall back into the well resulting in rapid changes in the pressure head within the well. If "rawhiding" is to be used, there cannot be a backflow prevention valve in the pump or eductor line. Another method of backwashing is to pump water into the well in sufficient volume to maintain a hydraulic head greater than that in the formation. Water used during this method will be of a known source and chemistry. The impact of foreign water on the local groundwater quality should be evaluated prior to proceeding and, added water should be removed by pumping after development is complete. A minimum of 3 volumes of water should be removed after backwashing with this technique in order to ensure that all added water within the formation has been eradicated. Prior to using this method, local, state, and federal guidance should be reviewed. Do not use this method in cases where the water pumped into the well is potentially contaminated.

6.1.4 Bailing

The use of dedicated disposable bailers is an effective way of manually developing small diameter wells that have a high static water table or are relatively shallow in depth [<4.6 m (20 ft)]. Use of a bailer that has a diameter close to that of the well screen is recommended in order to sufficiently agitate sediments that have settled at the bottom of the well. The bailer should be operated throughout the screened interval. Bottom loading bailers can extract sediment that has settled to the bottom of the well by short rapid up and down motions of the bailer at the bottom of the well which stir up the settled sediments and collect the particulates in the bailer. Bailers used for development should never be left inside the well after development is complete.

6.2 Development Procedures

The development of monitoring wells will be initiated no sooner than 48 hours after or no later than 7 days after the final installation of the monitoring well. For well development by pumping, water from the entire water column will be while periodically raising and lowering the pump intake in order to agitate sediments within the well. Well development will be completed a minimum of 14 days prior to any groundwater sampling occurs. Well development by pump procedures includes:

1. Open and record the condition of the well head in the Field Logbook.
2. Check for volatile organic compounds with a PID immediately after opening the well head and record the reading in the Field Logbook.

3. Measure the depth to the static groundwater level using a groundwater interface probe, or similar, in the well before beginning development. Record the value to the nearest 0.01 foot in the Field Logbook and the Well Development Form. Depth should be recorded from a pre-determined point on the well (typically a notch in the PVC riser).
4. Measure the total depth of the well prior to development by lowering a groundwater interface probe, or similar, to the base of the well. Depth should be recorded from a pre-determined point on the well (typically a notch in the PVC riser). Record the value to the nearest 0.1 foot in the Field Logbook and Well Development Form.
5. Connect the appropriate tubing, electrical supply lines and safety line to the pump and lower the pump into well. The pump should always remain completely submerged in groundwater.
6. Ensure that the effluent tubing connected to the pump is secured in the waste water receptacle (i.e. plastic tote, 55-gallon drum, 5-gallon bucket).
7. Energize the pump and adjust the pumping rate so as to minimize spillage from the effluent tubing to the waste water receptacle. Record the flow/pumping rate.
8. Development will be considered complete when the well water is clear to the unaided eye and a minimum of three times the standing volume of the well (to include the well screen, casing, plus saturated annulus, assuming 30% porosity for the annulus) has been removed.
9. Should the recharge be so slow that the required volume cannot be removed in 48 consecutive hours of development, the water remains discolored, or excess sediment remains after the three to five volume removal; contact the Project Manager for guidance.
10. Measure the total depth of the well after development and the total volume of water removed from the well. Record the value to the nearest 0.1 foot in the Field Logbook and the Well Development Form.

6.2 Development Records

The following information should be recorded in the Field Logbook and/or Well Development Form during development:

1. Date and time of development
2. Start and stop time of development
3. Static water level in the well prior to development
4. Total depth to the bottom of well
5. Volume of water column in well
6. Volume of water to be evacuated for development
7. Description of visual quality of water being removed, including color, odor, turbidity and volume of water removed when the observations were made. At a minimum, a brief description of the water removed should be made after initially starting development and after each well volume of water is removed.
8. Type and size of pump and/or bailer used
9. Description of surge techniques
10. Pumping rate and any changes to the rate after initially setting the pump rate
11. Total volume of water removed.

7.0 REFERENCES

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 1994. *Monitor Well Design, Installation, and Documentation at Hazardous and/or Toxic Waste Sites*. EM 1110-1-4000. November.

United States Environmental Protection Agency (USEPA), 1991. *Handbook of Suggested Practices for the Design and Installation of Ground Water Monitoring Wells*. EPA160014-891034. March.

United States Environmental Protection Agency (USEPA), 1992. *RCRA Ground Water Monitoring: Draft Technical Guidance.* November

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

Surging - Surging is a process in which a plunger-type device called a surge plunger or surge block, is moved up and down within the well screen to force groundwater to alternately flow in and out through the sand pack. This back and forth movement of water facilitates removal of fines from the formation immediately adjacent to the well while preventing bridging (wedging) of sand grains.

9.0 ATTACHMENTS

Well Development Log

GROUNDWATER MONITORING WELL DEVELOPMENT FORM

Client:	Sampling Organization: Earth Resources Technology
Site Name:	Sampler (s):
Project No.:	Screen Interval (feet below ground surface):
Date:	Pump Intake (feet below ground surface):
Well Number:	Purging Device (Pump Type):

Well	PID	DTW	TD	WV	MPV	Start Time	End Time	APV	Observations

PID = photo-ionazation detector

DTW = depth to water (in feet), from TOC

TD = total water depth (in feet), before development/after development

WV = well volume (gallons)

MPV = minimum purge volume (gallons)

APV = actual purge volume (in gallons)

Earth Resources Technology, Inc. Standard Operating Procedure **WELL PURGING – PUMPING METHOD**			
Effective Date: **01/22/09**	Version: **001**	**ERT SOP-05-6**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
Signature	1/23/09 Date	Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide general reference information on well purging by a high volume pumping method prior to environmental sampling of groundwater wells. The methods and equipment described are for the purging of water samples from the saturated zone of the substrata.

2.0 SCOPE

This procedure applies to purging relatively large volumes of water in a shallow to medium depth well (typically up to 75 feet in total depth).

3.0 REQUIREMENTS

The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the monitoring well and to avoid physical or chemical alteration of the water due to purging and sampling techniques. Typically, in between sampling events there will be little or no vertical mixing of stagnant water in the well and stratification will occur. The well water in the screened section may mix with groundwater due to normal hydraulic flow, but stagnant water above the screened section will remain isolated.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that appropriate well purging procedures are established in the project-specific Sample and Analysis Plan and that the field personnel assigned to the activity are properly trained and briefed.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that the well purging techniques utilized during field activities are in accordance with this procedure and the project-specific Sample and Analysis Plan. When purging is complete, the Field Team Leader is responsible for reviewing data entered into the Filed Logbook and/or the Well Purge Data Forms for accuracy and completeness. Upon completion of this review, the Field Team Leader will transfer custody of the forms and Field Logbook to the Project Manager

4.3 Field Team

The Field Team is responsible for purging wells in accordance with this procedure and the project-specific Sample and Analysis Plan. The Field Team will be responsible for purging

wells, performing appropriate physical measurements and observations, recording observations within the Field Logbook and/or Purge Data Sheets and containment of purged water. The Field Team will record pertinent information including amount of water purged, pH, specific conductivity, temperature, and turbidity on the Well Purge Data Form for each well purged. These parameters will be recorded as qualitative data and will not be used for determination of sampling interval.

5.0 EQUIPMENT

The following equipment shall be available for well purging via the pumping method:

- Submersible Centrifugal Pump
- Power Source with GFCI
- Chemically Inert Tubing
- Electronic water level meter (graduated to 0.01 feet)
- Multi-Parameter Water-Quality Meter
- Nitrile gloves
- Photoionization Detector
- 5 gallon receptor buckets for temporary purge water containment
- DOT liquid rated drums for containerization of purge water
- Well Purge Data Form
- Field Logbook
- Graduated vessel
- Timing device (stopwatch)
- Calculator

6.0 PROCEDURE

General

Purge volumes for each well will vary depending on the intent of the monitoring program and the local hydrogeologic conditions.. A well will not be sampled until the purging procedure has been followed to completion.

Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to determine the volume of standing water in the well column. Calculations shall be recorded in the Field Logbook and the Well Purge Data Forms.

1. If possible, obtain all available information on well construction (location, casing, screens, etc.).
2. Measure and record static water level (depth below ground level or top of casing reference point), per ERT SOP-07-2. If the total well depth is unknown, determine total depth of well using a clean, decontaminated, water level indicator.
3. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
4. Calculate the volume of water in the casing

Diameter of Casing or Hole (in)	Gallons per foot of depth	Cubic feet per foot of depth
2	0.163	0.0218
4	0.653	0.0873
6	1.469	0.1963

Water column volume (gallons) = (TD – DTW) x gallons per foot of depth

Specific Procedure

- Field personnel must always use new nitrile gloves.
- When known, purging and sampling order for multiple wells should proceed from least impacted to most impact in order to limit the potential for cross-contamination between monitoring wells or transfer of constituents.

1. Measure the background for volatile organic compounds (VOCs) using a photoionization detector (PID) then open the well and screen the well head with the PID. If volatile organic compound (VOC) concentrations are equal to or greater than 1000 parts per million (ppm), immediately recap the well and inform the Project Manager. Record the measured PID reading in the Field Logbook and/or the Well Purge Sheet.

   Monitoring head space VOC concentrations is a health and safety requirement and should be instituted at the discretion of the Field Manager and the requirements of the Site Health and Safety Plan.
2. Measure the depth to water in the well per ERT SOP0-7-2 and record the data in the Field Logbook and/or Well Purge Data Form.
3. Calculate the volume of water in the well. Record this information in the Field Logbook and/or Well Purge Data Form. A minimum of three well volumes will be purged from groundwater monitoring wells prior to sampling unless a well is purged dry. If purging results in drying the monitoring well, the well should be sampled as soon as sufficient volume of groundwater has recovered in the well. Note in the Field Logbook and/or Well Purge Data Form that the well was purged dry and indicate the volume of water removed.
4. Lower the purge pump into the well until it is completely submerged.
5. Place the pump effluent tubing into the receptor container and start the pump in accordance with the pump's operation manual. Record pump rates (if sustainable) and the total volume of water purged from the well.
6. Water quality parameters should be measured at the start of the purging process, after each well volume is removed, and prior to the collection of groundwater samples (minimum of 4 data measurements should be recorded in the Field Logbook and/or the Well Purge Data Sheets) to acquire qualitative data that will help evaluate geochemical characteristics of the aquifer. Measured groundwater quality data should be entered into the Field Logbook and/or Well Purge Data Form.
7. Groundwater samples shall be collected immediately after purging is complete in accordance with ERT SOP-06-5.

8. Whenever the receptor container is three quarters full, the purge water should be transferred to a DOT approved liquids drum, or similar large container, for containerization per ERT SOP-06-4 or disposed of in accordance with the project-specific Sample and Analysis Plan.
9. Carefully withdraw the purge pump from the well after purging is complete, decontaminate the pump in accordance with ERT SOP-03-1
10. Dispose of all investigative derived waste (IDW) items in accordance with ERT SOP-06-4 and the project-specific Sample and Analysis Plan.

7.0 REFERENCES

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 1992. *RCRA Ground Water Monitoring: Draft Technical Guidance.* November.

United States Environmental Protection Agency (USEPA), 2002. *Ground-Wager Sampling Guidelines for Superfund and RCRA Project Managers*. Ground Water Forum Issue Paper, EPA/542/S-02/001. May.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York.

8.0 DEFINITIONS

None

9.0 ATTACHMENTS

Well Purge Data Form

WELL PURGE DATA FORM

SITE: _____ FIELD PERSONNEL: _____

DATE: _____ _____

WEATHER:

WELL DEPTH: _____ STICK-UP / FLUSH MOUNT

MONITORING WELL: _____ WELL SIZE: _____ Inches WELL VOLUME: _____ GAL

2 inch = 0.163 gal/ft 4 inch = 0.653 gal/ft 3 X WELL VOLUME: GAL

PID/FID READINGS BACKGROUND: _____ PUMP INTAKE DEPTH: _____ ft below TOC

BENEATH WELL CAP: DEPTH OF WATER BEFORE PUMP INSTALLATION: ft below TOC

TIME	PURGING	SAMPLING	pH (pH units)		SPECIFIC CONDUCTIVITY (mS/cm)		TURBIDITY (NTU)		DISSOLVED OXYGEN (mg/l)		TEMPERATURE (degrees C)		OXYGEN REDUX POTENTIAL (mv)		PUMPING RATE (ml/min)	DEPTH TO WATER (ft below toc)
			READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE		
				NA		NA		NA		NA		NA		NA		

COMMENTS:

TOTAL GAL PURGED:

Earth Resources Technology, Inc. Standard Operating Procedure **MONITORING WELL AND BOREHOLE ABANDONMENT**			
Effective Date: **01/26/09**	Version: **001**	**ERT SOP-05-7**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/26/09 Date	 Signature	1/26/09 Date

1.0 PURPOSE

The purpose of this procedure is to describe, in general terms, the principles and methods of properly abandoning a constructed monitoring well or borehole.

2.0 SCOPE

This procedure applies specifically to the abandonment of monitoring wells and boreholes with the understanding that compliance with federal or local regulation may require additional actions. Project specific procedural details associated with abandonment will be detailed in the project-specific Sample and Analysis Plan.

3.0 REQUIREMENTS

In order to minimize the potential migration of groundwater and soil constituents, at a minimum, the following procedures will be conducted in order to properly abandon intrusions into the subsurface. Abandonment may occur when a borehole or monitoring well has been damaged to such an extent that it is no longer functional, or in the case that a borehole or monitoring well has served it functional purpose and is no longer required. ERT will notify the client of intent to abandon intrusions, as appropriate. ERT primary responsibility will be for the oversight of the abandonment. ERT personnel will properly document the activity within the project Field Logbook. All abandonment records will become part of the permanent project files and any necessary abandonment forms will be forwarded to the applicable authority. A properly licensed driller will be retained for the abandonment or boreholes and wells as outlined below.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that all procedures for properly abandoning a borehole or monitoring well are detailed in the project-specific Sample and Analysis Plans and those activities associated with borehole and monitoring well abandonment are conducted in accordance with the Project Plans and local, state and federal regulations.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that monitoring wells and boreholes are abandoned in accordance with this procedure and applicable project-specific requirements.

4.3 Field Team

The Field Team is responsible for ensuring that borehole and monitoring well abandonment is conducted in accordance with this procedure and applicable project-specific requirements. The Field Team will document all activities within the Field Logbook.

4.4 Contracted Licensed Driller

The contracted licensed driller will be responsible for the mechanical abandonment of bore holes and wells and certifying the completion of abandonment. Abandonment will be conducted within all applicable municipal, state, and federal regulations.

5.0 EQUIPMENT

- Drilling Rig Equipped with Appropriate Drilling Tools
- Cement, Sand, Bentonite Powder, Bentonite Pellets, or Commercial Hole-Sealing Products

6.0 PROCEDURE

Prior to abandoning a monitoring well in place, all attempts will be made to mechanically remove the monitoring well construction from the borehole. If an abandoned well cannot be removed by mechanical means, it may be grouted with the well screen and casing in place. Each boring or well to be abandoned/decommissioned will be sealed by grouting from the bottom of the boring/monitoring well to the ground surface. Grout will be mixed in a container and consist of cement, water and three to five percent (by weight) bentonite powder. Mixed grout will be introduced by extending a tremie pipe to the bottom of the boring/well (i.e., to the maximum depth drilled/bottom of well screen) and using a mechanical pump, transferred from the mixing container through the tremie pipe and into the boring/well. Grout will be introduced in this fashion until undiluted grout completely fills the boring/well void space. All openings or ungrouted portions of the annular space(s) between an innermost well casing and borehole will be completely filled with grout.

When possible after 24 hours, the Field Team Leader will check the abandoned site for grout settlement. If settlement has occurred, the depression will be filled with grout and rechecked after another 24 hours has passed. Additional grout will be added per the previously described method except that the tremie pipe will be inserted to the top of the competent grout surface. This process will be repeated until firm grout extends from the base of the borehole/well to the ground surface.

Hand-augered boreholes, generally less than 10 feet in total depth may be abandoned using dry flaked bentonite chips and do not require the services of a licensed driller for abandonment. Betonite will be used to fill the entire borehole to ground surface. In order to adequately homogenize the hydrated the betonite chips, betonite should be introduced in 2 foot lifts and then hydrating using water. A mixing rod will then be used to ensure an event consistency throughout the lift. Once consistency is ensured, the next two foot lift of dry flaked betonite chips may be added. This process will be repeated until the grout mixture extends to the ground surface. When possible after 24 hours, the Field Team Leader will revisit the borehole and if settling has occurred, grout will be added as previously described. In some cases, dependent on the

constituents and/or characterization of the auger cuttings, collected cuttings from a hand-augered borehole may be replaced back to the hole.

7.0 REFERENCES

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 1994. *Monitor Well Design, Installation, and Documentation at Hazardous and/or Toxic Waste Sites*. EM 1110-1-4000. November.

United States Environmental Protection Agency (USEPA), 1991. *Handbook of Suggested Practices for the Design and Installation of Ground Water Monitoring Well*. EPA160014-891034. March.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

Borehole - For purposes of this procedure only, any hole drilled into the subsurface for the purpose of identifying lithology or installing monitoring wells.

Contaminant - Any substance, which if introduced, would degrade the quality of groundwater.

Grout - A slurry of cement, clay, or other material impervious to and capable of preventing movement of water. Typically a neat cement grout containing three to five percent bentonite powder by weight.

Earth Resources Technology, Inc. Standard Operating Procedure **SURFACE WATER AND SEDIMENT SAMPLING**			
Effective Date: **01/22/09**	Version: **001**	**ERT SOP-06-1**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
Signature	1/22/09 Date	Signature	1/21/09 Date

1.0 PURPOSE

The purpose of this procedure is to define the requirements necessary for environmental surface water and sediment sampling. This procedure describes the methods and equipment commonly used for collecting environmental samples of surface water and sediment samples for either onsite examination and testing, or for laboratory analysis.

2.0 SCOPE

Surface water and sediment sampling may be conducted at any site which contains surface drainage, water bodies (i.e. streams, lakes, ponds) or sites located hydraulically down-gradient from surface drainage sites. The collection of concentrated sludge or waste characterization samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein. Consequently, specific sampling requirements may be required. Any such modification to these procedures will be specified in the project-specific Sampling and Analysis Plan (SAP).

3.0 REQUIREMENTS

Many factors must be considered in developing a sampling program for surface water or sediments, including study objectives; accessibility; site topography, flow, mixing, and other physical characteristics of a water body; point and diffuse sources of contamination; and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on the vertical and lateral mixing within the body of water. For sediments, dispersion depends on bottom current or flow characteristics, sediment characteristics (density, size) and geochemical properties (which affect an adsorption/desorption). The project team developing the sampling plan must consider the mixing characteristics of the water bodies and also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that the procedures described in the project-specific Sample and Analysis Plan, at a minimum meet the requirements set forth in this procedure and those detailed in applicable federal and state guidance documents, and that the field team conducting the sampling are adequately trained and briefed on the prescribed methodology.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that surface water and sediment sampling is conducted in accordance with the procedures outlined in this procedure and in the project-specific Sample and Analysis Plan. At a minimum, this may include reviewing the Sample and Analysis Plan for project specific requirements, ensuring that the sampling team is briefed and trained on the project specific requirements, and overseeing the collection and management of the environmental samples.

4.3 Field Team

The Field Team is required to adhere to the sample collection requirements described in this procedure and the project-specific Sample and Analysis Plan. Deviations from these documents must first be consulted with the Field Team Leader and Project Manager. The Field Team is required to properly manage the sample collection and handling process of samples from the point of sample collection to relinquishment of samples to the analytical laboratory or designated courier.

5.0 EQUIPMENT

- Sample Containers
- Thermometer
- Stainless Steel Bowl and Spoon
- Stainless Steel Hand Auger, Shovel, or Spoon
- Filtering Equipment (if analyzing for metals in water)
- Open Tube
- Dip Sampler
- Kemmerer or Van Dorn Sampler
- Hand-Driven Tube Sampler with Liners (Brass or Stainless Steel)
- Decontamination Equipment and Supplies
- Pump

6.0 PROCEDURE

General Procedures

The following section outlines general procedures for collecting surface water and sediment samples. All sampling equipment should be cleaned and decontaminated prior to use in accordance with ERT SOP-03-1.

- Unless specified in the project-specific Sample and Analysis Plan, when sampling surface water and sediments from the same location, surface water samples will always be collected first and sediments second in order to minimize disturbance of sediments and unintentional collection of these disturbed sediments in the surface water samples..
- Samples will always be collected first at the most down-gradient location and progress to the furthest up-gradient location in order to minimize potential cross-contamination or constituent transfer from one sample location to another.

- Sample volumes at each location will first be collected for volatile organic compound (VOC) analysis followed by sample volumes collected for non-volatile organics and inorganics analysis.
- Prior to collecting sediment and/or surface water samples, the sampling devise shall be decontaminated in accordance with ERT SOP-03-1 Twigs, leaves, pebbles, and debris that are not components of the matrix of interest will be properly removed by the Field Team using nitrile gloves or stainless steel tongs.
- Sediment sample volumes collected for VOC analysis will be collected directly from the sampler and immediately placed in the dedicated sample container and sealed in order to minimize volatilization. The remaining sample volumes to be collected for analysis will be composited in a stainless-steel bowl and allocated to the dedicated sample containers. Duplicates and split samples shall be collected at the same time as the original sample and in the same analytical sequence.
- Surface water sample volumes collected for VOC analysis will be collected ensuring that no bubbles or air space are trapped in the dedicated volatile organic analysis (VOA) sample container. Sediment sample volumes collected for VOC analysis will be sufficient to completely fill the dedicated sample container eliminated, when possible, all voids or head space. Sediment sample volumes should be decanted, as possible, to eradicate excess water.
- Prior to and after sample volumes are collected at each location, the sampling equipment will be decontaminated in accordance with ERT SOP-03-1.
- As required in the project-specific Sample and Analysis Plan, sample volumes collected for dissolved inorganic parameters in surface waters may be field filtered in accordance with ERT SOP-06-8 or laboratory filtered prior preservation. Surface water sample volumes collected for total inorganic analysis will not be filtered.

Water Sampling Techniques

Surface water sample volumes collected for VOC analysis will first be collected in dedicated unpreserved sample containers and then immediately transferred to dedicated VOA sample containers with Teflon® septa or other nonreactive material and the prescribed volume of hydrochloric acid preservative. Generally, the VOA sample container will be provided by the analytical laboratory containing the prescribed volume of hydrochloric acid preservatives. Preserved samples have a two week holding time, whereas, unpreserved samples have only a seven day holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles rendering the sample unacceptable for VOC analysis. In this case, unpreserved vials should be used and arrangements confirmed with the laboratory prior to sampling in order to ensure acceptable receipt of the unpreserved sample volumes.

Dip Sampling

A sample may be collected directly into the sample container when unpreserved dedicated sample containers are being utilized. Surface water volumes collected for analysis requiring preservation will be collected in a dedicated unpreserved sample container and immediately transferred into a dedicated sample container with the prescribed sample preservative volume. In

most cases, the analytical laboratory will provided dedicated sample containers with the prescribed sample preservative pre-aliquotted. Whenever possible, surface water samples should be collected away from the shore line in order to minimize sediment infiltration into the sample volume. If the water body is not stagnant (i.e. stream, river) the sampler should face upstream while collecting the appropriate sample volume and minimize disturbing the bottom sediments.

Weighted Bottle Sampling

A grab sample can also be taken using a weighted holder that allows a sample to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling within a specific interval within the water column. Several of these samples can be combined to provide a vertical composite. Alternatively, in many cases it is acceptable to use a dedicated sample container to obtain a vertical composite sample. The dedicated sample container can be lowered to the bottom of the desired interval and raised to the top of the desired interval at a uniform rate

A closed weighted bottle sampler consists of a stoppered glass or plastic bottle, weight and/or holding device, and lines to open the stopper and to lower or raise the bottle. The proper procedure for sampling using this equipment is:

1. Gently lower the closed weighted sampler to the desired depth. Do not prematurely remove the stopper. Air bubbles forming at the surface of the water bottle may indicate that the stopper has opened prematurely. If air bubbles are observed, the sampler should be inspected prior to collecting the sample volume.
2. Disengage the stopper with a sharp tug of the sampler line.
3. Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
4. Raise the sampler and cap the bottle.
5. Preserve sample, if required, with appropriate preservatives or transfer the sample volume to the dedicated sample container.
6. Decontaminate weighted sample bottle in accordance with ERT SOP-03-1 if it will be used for further sample collection.

Pumps

Pumps which operate by a bellows, diaphragm, or siphon action should not be used to collect samples which will be analyzed for volatile organics because the slight vacuum applied may induce volatilization. In order to avoid contamination of the pump, a liquid trap consisting of a vacuum flask or other vessel to collect the sample should be inserted between the sample inlet hose and the pump.

Tubing used for the inlet hose shall be nonreactive (preferably Teflon®). The tubing and liquid trap must be thoroughly decontaminated between uses, per ERT SOP-03-1, or disposed of after a single use.

When sampling, the tubing is weighted and lowered to the desired depth. The sample is then obtained by operation of the pump and subsequently transferred from the trap to the dedicated sample container.

Kemmerer/Van Dorn Samplers

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon® coated sampler, a Standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends open while being lowered in a vertical position to allow free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In either case, a "messenger" is sent down the line when the sampler is at the designated depth which causes the rubber stoppers to close, thereby allowing the sample to be contained and raised. The sample is removed through a valve to fill the sample containers.

Sediment Sampling Techniques

Sediment samples are typically collected at the same locations or adjacent to surface water locations. Typically if only one sediment sample is to be collected, the sample locations will be approximately at the center of the water body. This is particularly true for reservoirs that are formed by the impoundment of rivers or streams. Generally, coarser grained sediments are deposited near the headwaters of the reservoir. Bed sediments near the center are generally composed of fine-grained materials which may, because of their lower porosity and greater surface area, contain greater concentrations of contaminants. The shape, flow pattern, depth distribution, and water circulation patterns must all be considered when selecting sediment sampling locations. In streams, areas likely to have sediment accumulation (i.e., bends, behind islands or boulders, quiet shallow areas or very deep, low velocity areas) may be sampled while areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials will be avoided. Refer to the project-specific Sample and Analysis plan for the appropriate sample locations for sediments and surface waters.

Scoop Samplers

Stainless steel scoops may be used to collect sediment samples. Scoops have a limited reach of approximately eight feet, if attached to an extension.

Dredge Samplers

A dredge is a vessel that is dragged across the bottom of the surface being sampled, collecting a composite of surface sediments and associated benthic fauna. This type of sampler is used primarily for collecting indigenous benthic fauna rather than samples for chemical analyses. Because the sample is mixed with the overlying water, no pore-water studies can be conducted utilizing dredged samples. Additionally, because the walls of the dredge are typically nets, they act as a sieve and mainly coarser material is trapped, resulting in the loss of fine sediments and water-soluble compounds. Associated sample washing may potentially bias sample results. Typically, sample analysis results from dredge sampling should be considered qualitative since it is difficult to determine the actual surface sampled by the dredge. For these reasons, dredge samplers are not recommended within this procedure.

Grab Samplers

Grab samplers have jaws that close by a trigger mechanism upon impact with the bottom surface. Grab samplers offer the advantage of being able to collect a large amount of material in one sample, but are highly unpredictable in determining sampling depth. Substantial contaminant variation with depth is unlikely in shallow channel areas without direct contamination sources, in areas with frequent ship traffic, or from sediments that are dredged at regular intervals. In these

situations, bottom sediments are frequently re-suspended and mixed, effectively preventing stratification. In such cases, surface grab samples represent the mixed sediment column.

Core Samplers

Core samplers are tubes that are inserted into the sediment by various means to obtain a cylinder or box sample of material at known depths. Corers can be simple, hand-operated devices used by scuba divers, or they can be large, costly, motor-driven mechanisms that can collect samples from great depths. Corers are recommended whenever sampling to depth is required, or when the variation in contamination with depth is of concern. Examples of corers are gravity corers, piston corers, vibra-corers, split- spoon cores, and box core samplers. The choice of corer design depends on factors such as the objectives of the sampling program, sediment volumes required for testing, sediment characteristics, water depth, sediment depth, and currents or tides. Refer to the project-specific Sample and Analysis Plan for the appropriate sample equipment specific to the project objectives

NOTE: The sample processing area may be located on the water vessel depending on the calmness of the sampling environment (e.g., no waves or current) and available workspace on the sampling platform (e.g., use of a large barge).

7.0 REFERENCES

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8.0 DEFINITIONS

Environmental Sample - Low concentration sample typically collected offsite and not requiring DOT hazardous waste labeling as a high hazard sample.

Hazardous Waste Sample - Medium to high concentration sample (e.g., source material, sludge leachate) requiring DOT labeling and Contract Lab handling as a high hazard sample.

Earth Resources Technology, Inc. Standard Operating Procedure **ENVIRONMENTAL SOIL SAMPLING**			
Effective Date: **01/27/09**	Version: **001**	**ERT SOP-06-03**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
	1/27/09		1/26/09
Signature	Date	Signature	Date

1.0 PURPOSE

The purpose of this procedure is to outline the general procedures for environmental soil sampling. Soil sampling aids in characterizing substrata, and the nature and extent of soil constituents. Soil samples may be collected at different locations within the horizontal and vertical plane in order to characterize the distribution of constituents within the media

2.0 SCOPE

Soil sampling is potentially applicable to any site which characterization of media constituents is necessary. A variety of sampling techniques are available for collection of soil samples. These include split-spoon sampling, collecting auger cuttings, Shelby tube sampling and continuous coring sampling. Split-spoon sampling is the most commonly used technique. The collection of internal quality control checks during soil sampling is specified in the project-specific Sample and Analysis Plan.

3.0 REQUIREMENTS

Soil sample collection points should be as close to possible to the proposed location. In most cases accuracy within one foot horizontally is adequate. Specific sampling requirements will be detailed within the project-specific Sampling and Analysis Plan (SAP). Sample locations will be recorded in the Field Logbook and can be accomplished by hand sketching a diagram with measured distances. Record keeping for specific sample locations may be conducted by professional surveys, geographical positioning systems or measuring the distance from site specific permanent/semi-permanent features. The precision of the soil sampling will be determined by the following data quality objectives:

- Sample collection information will be recorded in the Field Logbook and/or Boring Log Form. This will include the sample locations, sample identification (ID), requested analysis and field observations regarding the sample collected.
- Surface/air contact may be minimized by placing the sample in an airtight container immediately after collection.
- Sampling and sample preparation equipment will be decontaminated in accordance with ERT SOP-03-01 prior to and after each sample is collected unless dedicated disposable equipment is used.
- Soil samples collected for volatile organic analytes (VOAs) will be collected and containerized undisturbed, if possible.

- Depth-profile sampling must comply with the above requirements. Care must be taken to prevent cross-contamination, transfer of constituents and misidentification of samples.
- Immediately after containerizing a sample, the sample for laboratory analysis will be labeled in accordance with the project-specific SAP.
- Vertical depth control tolerances are specified in the project-specific Sample and Analysis Plan.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that the procedures described in the project-specific SAP, at a minimum meet the requirements set forth in this procedure and those detailed in applicable federal and state guidance documents, and that the field team conducting the sampling are adequately trained and briefed on the prescribed methodology**.**

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that soil sampling is conducted in accordance with the procedures outlined in this procedure and in the project-specific SAP. At a minimum, this may include reviewing the SAP for project specific requirements, ensuring that the sampling team is briefed and trained on the project specific requirements, and overseeing the collection and management of the environmental samples.

4.3 Field Team

All members of the Field Sampling Team are responsible for implementing the sampling procedures, including the classification soil and rock samples, collecting soil and rock samples, packing and sealing of soil and rock samples. Such duties may be performed by geotechnical engineers, field technicians, or other qualified field personnel.

5.0 EQUIPMENT

The following pieces of equipment may be needed for the collection of depth-specific soil samples:

1. Drilling Equipment (i.e. mud rotary drill)
2. Split-Spoon Sampling Equipment (typically either 1 3/8" or 2 1/2" I.D)
3. Shelby Tube Sampling Equipment
4. Direct Push Machine (Geoprobe 6620® or similar)
5. Stainless Steel Hand Auger
6. Shovel, or Post-Hole Digger
7. Stainless Steel Trowel, Spoon, or Bucket Auger
8. Teflon® or Stainless Steel Spatula
9. Stainless Steel Bowl or Teflon® Mixing Board
10. Organic Vapor Monitoring Device (PID)
11. Appropriate Sampling Containers
12. Ziploc-type plastic bags
13. Decontamination Supplies
14. Field Logbook and Field Sampling Forms
15. Knife or chemically inert spatula type instrument
16. Nitrile Gloves

6.0 GENERAL PRECAUTIONS

The following general precautions should be taken when sampling:

- A clean pair of new, disposable nitrile gloves shall be worn at each sampling location and gloves should be donned immediately prior to sampling.
- All work is to be conducted on a clean surface.
- Sample collection, preservation, and method-specific handling procedures outlined in the project-specific SAP will be used for samples collected for laboratory analysis.
- To prevent cross-contamination between samples, all sample containers for a sampling location should be sealed in dedicated plastic bags, especially when the sampled medium is suspected of containing high concentrations of volatile organics.
- Samples of waste or highly contaminated samples should never be placed in the same storage container (ice chest) as environmental samples.
- Wherever possible, one member of the field team should be dedicated to recording all the collection notes, fill out sample labels, field sheets, etc., while the other members collect the required sample volumes. This is especially important when subjective decisions and descriptions are being made.
- Sample collection activities should precede progressively from the suspected least impacted area to the suspected most impacted area.
- Field personnel should take precautions to prevent cross-contamination from sampling equipment. Stainless steel samplers are preferable. All samplers should be properly decontaminated and inspected for visible signs of deterioration before each use or be dedicated and disposed of after each use.
- Adequate field control samples should be collected. Typically field control samples consist of Field Blank Samples and Equipment Blank Samples. Consult the project-specific SAP for details.

7.0 PROCEDURES

7.1 Manual Soil Sampling Methods

General

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches, however the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12-inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical.

Spoons
Stainless steel spoons may be used for surface soil sampling to depths of approximately 6-inches below ground surface where conditions are generally soft and non-indurated and there is no problematic vegetative layer to penetrate.

Special Considerations When Using Spoons

- When using stainless steel spoons, consideration must be given to the procedure used to collect the volatile organic compound sample.
- When compositing, make sure that each composite location (aliquot) consist of equal volumes, i.e., same number of equal spoonfuls.
- If a thick, matted root zone is present at or near the surface, it should be removed before the sample is collected

Hand Augers
Hand augers may be used to advance boreholes and collect soil samples in the surface and shallow subsurface intervals. Typically, 4-inch stainless steel auger buckets with cutting heads are used. The bucket is advanced by simultaneously pushing and turning using an attached handle.

Surface Soil Sampling
When conducting surface soil sampling with hand augers, the auger buckets may be used with a handle alone or with a handle and extensions. The bucket is advanced to the appropriate depth and the contents are transferred to the homogenization container for processing.

Subsurface Soil Sampling
Hand augers are the most common equipment used to collect shallow subsurface soil samples. Auger holes are advanced one bucket at a time until the sample depth is achieved. When the sample depth is reached, the bucket used to advance the hole is removed and decontaminated following ERT SOP03-1. The clean auger bucket is then placed in the hole and filled with soil to make up the sample and removed. The practical depth of investigation using a hand auger depends upon the soil properties and depth of investigation. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow or collapse. Hand augers may also be of limited use in tight clays or cemented sands. In these soil types, the greater the depth attempted, the more difficult it is to recover a sample due to increased friction and torqueing of the hand auger extensions. At some point these problems become so severe that power equipment must be used.

Special Considerations for Soil Sampling with the Hand Auger

- Because of the tendency for the auger bucket to scrape material from the sides of the auger hole while being extracted, the top several inches of soil in the auger bucket should be discarded prior to placing the sample in the required sample container.

- Power augers, such as the Little Beaver®, and drill rigs may be used to advance boreholes to depths for subsurface soil sampling with the hand auger. They may not be used for sample collection. When power augers are used to advance a borehole to depth

for sampling, care must be taken that exhaust fumes, gasoline and/or oil do not contaminate the borehole or area in the immediate vicinity of sampling.

7.2 Direct Push Soil Sampling Methods

General
These methods are used primarily to collect shallow and deep subsurface soil samples. Three methods are available for use with either the Geoprobe® or the drill rig adapted with a hydraulic hammer. All methods involve the collection and retrieval of the soil sample within a thin-walled liner. The following sections describe each of the specific sampling methods that can be accomplished using direct push techniques, along with details specific to each method.

Large Bore® Soil Sampler
The Large Bore® (LB®) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of depth-discrete subsurface soil samples. The sample barrel is approximately 30-inches (762 mm) long and has a 1.5- inch (38 mm) outside diameter. The LB® sampler is capable of recovering a discrete sample core 22 inches x 1.0 inch (559 mm x 25 mm) contained inside a removable liner. The resultant sample volume is a maximum of 283 ml. After the LB® sample barrel is equipped with the cutting shoe and liner, the piston-rod point assembly is inserted, along with the drive head and piston stop assembly. The assembled sampler is driven to the desired sampling depth, at which time the piston stop pin is removed, freeing the push point. The LB® sampler is then pushed into the soil a distance equal to the length of the LB® sample barrel. The probe rod string, with the LB® sampler attached, is then removed from the subsurface. After retrieval, the LB® sampler is then removed from the probe rod string. The drive head is then removed to allow removal of the liner and soil sample.

Macro-Core® Soil Sampler
The Macro-Core® (MC®) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of either continuous or depthdiscrete subsurface soil samples. Although other lengths are available, the standard MC® sampler has an assembled length of approximately 52 inches (1321 mm) with an outside diameter of 2.2 inches (56 mm). The MC® sampler is capable of recovering a discrete sample core 45 inches x 1.5 inches (1143 mm x 38 mm) contained inside a removable liner. The resultant sample volume is a maximum of 1300 ml. The MC® sampler may be used in either an open-tube or closed-point configuration. Samples collected for chemical analyses must be collected with the closed-point configuration. If used for collection of soil for stratigraphic descriptions, the open-tubed configuration is acceptable.

Dual Tube Soil Sampling System
The Dual Tube 21 soil sampling system is a direct push system for collecting continuous core samples of unconsolidated materials from within a sealed outer casing of 2.125-inch (54 mm) OD probe rod. The samples are collected within a liner that is threaded onto the leading end of a string of 1.0-inch diameter probe rod. Collected samples have a volume of up to 800 ml in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core. Use of this method allows for collection of continuous core inside a cased hole, minimizing or preventing cross-contamination between different intervals during sample collection. The outer casing is advanced, one core

length at a time, with only the inner probe rod and core being removed and replaced between samples. If the sampling zone of interest begins at some depth below ground surface, a solid drive tip must be used to drive the dual tube assembly and core to its initial sample depth.

Special Considerations When Using Direct Push Sampling Methods

- *Liner Use and Material Selection* – Due to the mode of operation, the samples must be collected with a liner. Liners are available in the following materials: stainless steel, brass, cellulose acetate butyrate (CAB), PETG, polyvinyl chloride (PVC) and Teflon®. The required liner material will be specified in the project-specific Sample and Analysis Plan.
- *Sample Orientation* – When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely the depth at which an aliquot was collected. Maintaining proper orientation is typically accomplished using vinyl end caps. Typically orientation is indicated by marking on the exterior of the liner with a permanent marker.
- *Core Catchers* – Occasionally the material being sampled lacks cohesiveness and is subject to crumbling and falling out of the sample liner. In cases such as these, the use of core catchers on the leading end of the sampler may help retain the sample until it is retrieved to the surface. Materials of construction for core catchers must be consistent with the type of liner used, i.e., if stainless steel liners are required, stainless steel core catchers must be used.

7.3 Split Spoon/Drill Rig Methods

General

Split spoon sampling methods are used primarily to collect shallow and deep subsurface soil samples. All split spoon samplers, regardless of size, are basically split cylindrical barrels that are threaded on each end. The leading end is held together with a beveled threaded collar that functions as a cutting shoe. The other end is held together with a threaded collar that serves as the sub used to attach the spoon to the string of drill rod. Two basic methods are available for use, including the smaller diameter standard split spoon, driven with the drill rig safety hammer, and the larger diameter continuous split spoon, advanced inside and slightly ahead of the lead auger during hollow stem auger drilling. The following sections describe each of the specific sampling methods, along with details specific to each method.

Standard Split Spoon

A drill rig is used to advance a borehole to the target depth. The drill string is then removed and a standard split spoon is attached to a string of drill rod. Split spoons used for soil sampling must be constructed of stainless steel and are typically 2.0-inches OD (1.5-inches ID) and 18-inches to 24-inches in length. Other diameters and lengths are common and may be used if constructed of the proper material. After the spoon is attached to the string of drill rod it is lowered into the borehole. The drill rig safety hammer is then used to drive the split spoon into the soil at the bottom of the borehole. After the split spoon has been driven into the soil, filling the spoon, it is

retrieved to the surface, where it is removed from the drill rod string and opened for sample acquisition.

Continuous Split Spoon
The continuous split spoon is a large diameter split spoon that is advanced into the soil column inside a hollow stem auger. Continuous split spoons are typically 3-inches to 5- inches in diameter and either 5-feet or 10-feet in length, although the 5-foot long samplers are most common. After the auger string has been advanced into the soil column a distance equal to the length of the sampler being used it is returned to the surface. The sampler is removed from inside the hollow stem auger and the threaded collars are removed. The split spoon is then opened for sampling.

Special Considerations When Using Split Spoon Sampling Methods

Always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the borehole wall after removal of the drill string prior to and during inserting the split spoon.

7.4 Shelby Tube/Thin-Walled Sampling Methods

General
Shelby tubes, also referred to generically as thin-walled push tubes or Acker thin-walled samplers, are used to collect subsurface soil samples in cohesive soils and clays during drilling activities. In addition to samples for chemical analyses, Shelby tubes are also used to collect relatively undisturbed soil samples for geotechnical analyses, such as hydraulic conductivity and permeability, to support hydrogeologic characterizations at hazardous waste and other sites.

Shelby Tube Sampling Method
A typical Shelby tube is 30-inches in length and has a 3.0-inch OD (2.875 ID) and may be constructed of steel, stainless steel, galvanized steel, or brass. They also typically are attached to push heads that are constructed with a ball-check to aid in holding the contained sample during retrieval. If used for collecting samples for chemical analyses, it must be constructed of stainless steel. If used for collecting samples for standard geotechnical parameters, any material is acceptable. To collect a sample, the tube is attached to a string of drill rod and is lowered into the borehole, where the sampler is then pressed into the undisturbed clay or silts by hydraulic force. After retrieval to the surface, the tube containing the sample is then removed from the sampler head. If samples for chemical analyses are needed, the soil contained inside the tube is then removed for sample acquisition. If the sample is collected for geotechnical parameters, the tube is typically capped, maintaining the sample in its relatively undisturbed state, and shipped to the appropriate geotechnical laboratory.

7.5 General Sample Handling for Intact Soil Cores

1. Expose or remove the core from the sampling device.
2. Use a knife or spatula type instrument to remove any slough or cuttings that may have fell into the hole above the sampling depth

3. Use a knife or spatula to cut the core lengthwise. Successive passes should be done until the core is cut halfway. The core is then pried apart the rest of the way revealing a fresh face not smeared by the knife.
4. Once split apart any layering or structures should be noted.
5. Collect and containerize the environmental sample for laboratory analysis following the methodology specified in the project-specific SAP.
6. Describe the samples and record the description on the Borehole Logging Form in accordance with ERT SOP-07-3..
7. If samples are collected for laboratory analysis verify that samples have been properly labeled and stored as specified in the project-specific SAP.
8. This sampling procedure is repeated at appropriate intervals in accordance with the project-specific SAP.
9. Soil IDW generated by soil sampling and drilling activities will be disposed of according to ERT SOP 06-4 as prescribed in the project-specific SAP.
10. All sampling equipment, including internal components, will be decontaminated prior to use, between sampling events, and prior to demobilization.

8.0 COMPOSTING AND SPLITTING SAMPLES

Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provide an average concentration of contaminants over a certain number of sampling points. Compositing dilutes high-concentration aliquots; therefore, detection limits should be reduced accordingly. If the composite area is heterogeneous in concentration and its composite value is to be compared to a particular action level, then that action level must be divided by the total number of aliquots making up the composite for accurate determination of the detection limit.

Splitting Samples

Splitting samples (after preparation) is performed when multiple portions of the same samples are required to be analyzed separately. Fill the sample containers simultaneously with alternate spoonfuls of the homogenized sample

9.0 REFERENCES

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10.0 DEFINITIONS

Auger Cuttings - Soil brought to the surface by the action of the augers as they are screwed into the ground.

Hazardous Waste Sample - Medium to high concentration sample (e.g., source material, sludge, leachate) requiring DOT labeling and Contract Lab handling as a high hazard sample.

Shelby Tube Sampler - A thin-walled metal tube is used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from two to five inches in outside diameter and 18 to 54 inches in length. A stationary piston device is included in the sampler to reduce sampling disturbance and increase sample recovery.

Split-Spoon Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. This device can be driven into resistant (consolidated) materials using a drive weight mounted on the drilling rig. A standard split-spoon sampler, used for performing standard penetration tests, is two inches in outside diameter and 1 3/8 inches in inside diameter. This standard spoon typically is available in two common lengths, providing either 20 inch or 26 inch internal longitudinal clearance for obtaining 18 inch or 24 inch long samples, respectively. A five-foot long split-spoon sampler is also available.

Continuous Core Sampler - A steel tube, which may be split in half and held together by threaded collars or may be one piece. The sampler is usually five or ten feet in length and three to five inches in diameter. This device may be driven ahead of hollow-stem augers or may be driven into the soil by vibrational and/or rotary action.

Earth Resources Technology, Inc. Standard Operating Procedure **INVESTIGATIVE DERIVED WASTE (IDW) MANAGEMENT**			
Effective Date: **01/29/09**	Version: **001**	SOP#: **ERT SOP-06-4**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/29/09 Date	 Signature	1/28/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide general and specific considerations and procedures for use in managing investigation derived waste (IDW) generated during the course of environmental site investigation activities.

2.0 SCOPE

The procedure is applicable to ERT personnel involved in managing IDW generated during environmental site investigations. This procedure includes guidance on what constitutes IDW, and how to properly minimize, identify, characterize, stage and dispose of IDW.

3.0 REQUIREMENTS

To help minimize health and safety hazards associated with handling IDW, common sense, good judgment, and compliance with the protocols of this procedure and associated federal and state Regulations are essential.

Proper safety precautions must be observed when managing IDW. Refer to the project-specific work plan and to ERT's Health and Safety Plan (HASP) for guidance on necessary safety precautions.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that all activities involving IDW are properly managed and executed in accordance with federal and state regulation. The Project Manager is also required to ensure that all field personnel involved with the management of IDW are properly trained and made aware of the associated hazards.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring all IDW management precautions detailed in this procedure and the project-specific work plans are followed. The Field Team Leader is also responsible for ensuring that the Field Sampling Team manages all IDW in accordance with applicable project-specific requirements, federal and state regulation. Prior to leaving a site after completing field activities for a given day, the Field Team Leader is responsible for properly labeling and securing all IDW to be staged onsite, or ensure that all IDW has been removed from the site.

4.3 Field Sampling Team

The Field Sampling Team is responsible for minimizing the amount of IDW generated for containerization, properly labeling and staging IDW, and for the overall safe conduct of drum opening, sampling, and labeling operations when IDW is containerized in drums.

If any unexpected results (i.e. environmental releases) occur, the Field Sampling Team must inform the Project Manager immediately. Together with the Health and Safety Officer and outside assistance, if necessary, the most prudent course of action will be acted upon.

4.4 Health and Safety Officer

The Health and Safety Officer is responsible for safety of all on-site operations, alerting the Field Sampling Team of any potentially unsafe conditions, and halting work if on-site personnel or off-site public health is threatened.

5.0 EQUIPMENT

- 55-Gallon Drums
- Roll-Off Dumsters
- Nitrile Gloves
- Contractor-Grade Plastic Bags
- Non-Hazardous Waste Labels
- Hazardous Waste Labels
- Sharpie Pens
- Drum Dolly
- Drum Opening Equipment (i.e. bung wrench)
- PID
- Personal Protective Equipment as specified in the project-specific work plan.
- Spill Control Equipment
- Containment Equipment
- 6-Mil Plastic Sheeting
- Soil and Water Sampling Equipment
- Field Logbook

6.0 PROCEDURES

6.1 Types of IDW

Materials which constitute IDW include, but are not limited to:

- Personnel protective equipment (PPE) - This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
- Disposable equipment and items - This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers, disposable bailers, disposable tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Soil cuttings from drilling and/or hand augering.

- Drilling mud or water used for mud or water rotary drilling.
- Groundwater obtained through well development or well purging.
- Cleaning fluids such as detergents and wash water.
- Packing and shipping materials.

Table 6-1 lists the types of IDW commonly generated during field investigations and the current disposal practices that should be followed. For the purpose of determining the ultimate disposition of IDW, it is typically distinguished as being either hazardous or non-hazardous. This determination is based on either clear regulatory guidance or subsequent laboratory analysis.

6.2 IDW Minimization

By implementing a flexible field investigation plan that utilizes real-time data, the Field Team can optimize sample locations and numbers of samples collected in order to adequately characterize a site. The use of a phased approach to site investigations can also result in less IDW generation and more accurate constituent delineation.

Early investigation phases may be nonintrusive and may be performed using geophysics or aerial photography. Subsequent phases may be performed with minimally intrusive techniques such as direct push technology (DPT).

For permanent monitoring wells requiring multiple rounds of investigation, utilization of dedicated sampling equipment can reduce IDW. Low flow sampling techniques can also be an effective way of minimizing the generation of purge water. This technique assures groundwater parameters stabilize prior to sampling (representative of the aquifer) and eliminates the need to remove multiple volumes of well groundwater prior to sampling.

Advance planning should be used not only to minimize the quantities of IDW generated, but also to avoid generation of RCRA regulated wastes and the generation of wastes subject to land disposal restrictions.

- Use of RCRA regulated solvents should be replaced by power washing, steam cleaning, or use of non-RCRA solvents whenever possible.
- Boring sizes should be as small as possible to accomplish the specific task.

6.3 IDW Containerization

Depending on the type of IDW and extent of contamination, IDW may be staged onsite for subsequent characterization and disposal in:

- Contractor bags – typical for non-hazardous PPE and disposable sampling equipment
- Steel 55-gallon DOT-approved drums – typical for low volumes of soil and purge water, potentially hazardous PPE and hazardous IDW
- Roll-off dumpsters – typical for large volumes of hazardous and non-hazardous soil

Table 6-1. Typical IDW and Appropriate Management

TYPE	HAZARDOUS	NON-HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to Field Office for disposal in dumpster.
PPE-Reusable	Decontaminate as per SESDPROC-205, if possible. If the equipment cannot be decontaminated, containerize in plastic 5- gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Decontaminate as per ERT SOP03-1
Soil Cuttings	Containerize in DOT-approved container with tight-fitting lid. Identify and leave onsite with permission of site operator, otherwise arrange with program site manager for testing and disposal. Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.
Groundwater	Containerize in DOT-approved container with tight-fitting lid. Identify and leave onsite with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.
Decontamination Water	Containerize in DOT-approved container with tight-fitting lid. Identify and leave onsite with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.
Disposable Equipment	Containerize in DOT-approved container or 5-gallon plastic bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.
Trash	NA	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.

*** These materials may be placed on the ground if doing so does not endanger human health or the environment or violate federal or state regulations.*

6.4 Management of Non-Hazardous IDW

Disposal of non-hazardous IDW will be addressed in the project-specific Sampling and Analysis Plan. Disposable non-hazardous IDW may be placed into municipal dumpsters. Disposal of non-hazardous IDW such as drill cuttings, purge or development water, decontamination wash water, drilling mud, etc., will be specified in the approved project-specific Sampling and Plan. These materials must be staged in dedicated containers and not be placed into general trash dumpsters. The minimum requirements for managing non-hazardous IDW include:

- Non-hazardous liquid and soil/sediment IDW will be containerized for proper disposal when possible immediately upon generations, but at a minimum, prior to concluding site activity for the day.
- Soap and water decontamination fluids and rinsates of such cannot be placed in any water bodies and must be collected and disposed of properly.
- The collection, handling and proposed disposal method must be specified in the approved study plan or QAPP.

6.5 Management of Hazardous IDW

Management and disposal of hazardous or suspected hazardous IDW will be specified in the approved project-specific Sampling and Analysis Plan. Hazardous IDW must be disposed as specified in USEPA regulations. These wastes must be treated and disposed of at an appropriately licensed waste treatment facility. IDW determined to be hazardous waste, must be properly contained and labeled. Hazardous IDW may be stored on the site for a maximum of 90 days before it must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to allow arrangements for proper containerization, labeling, transportation and disposal/treatment in accordance with USEPA regulations. Most routine field activities will not produce hazardous IDW.

At a minimum, all hazardous IDW must be containerized and secured prior to leaving the site on a given day. Proper handling and disposal will be arranged prior to commencement of field activities and detailed in the project-specific Sampling and Analysis Plan.

6.6 Labeling Waste Containers

Immediately upon containerizing IDW, the container will be labeled with generator information, emergency contact information, IDW source and applicable characterization (non-hazardous or hazardous). If the IDW must be sampled in order to complete characterization, the container will be labeled as pending characterization using a non-hazardous label, with applicable generator and emergency contact information. Upon complete characterization, containerized IDW will be labeled with updated applicable information and appropriate non-hazardous label (Attachment 1) or hazardous label (Attachment 2). If it is determined by analytical procedure that the waste contained is hazardous, then a hazardous waste label shall be immediately filled out to completion and affixed to the container replacing the nonhazardous placard.

6.7 IDW Characterization

After the IDW has been containerized, it must be characterized in order to determine what disposal methods are applicable. Generally, PPE and other materials used for environmental sampling (i.e., dedicated tubing, dedicated bailers, nitrile glove) will not need to be characterized via laboratory analytics. If analytical sampling requirements are required, requirements will be

detailed in the project-specific Sampling and Analysis Plan. Characterization should determine the following:

- Is the IDW a RCRA hazardous waste?
- Is the IDW a hazardous or toxic substance under any other statute such as CERCLA, CWA, etc.?
- Is the IDW a non-hazardous?

Determining whether IDW constitutes RCRA hazardous waste will determine if:

- Land disposal restrictions apply
- The waste can be managed onsite
- The waste must be disposed of in a RCRA Subtitle C (hazardous waste) facility
- The waste can be disposed of in a RCRA Subtitle D (nonhazardous waste) facility.

Characterization involves sampling, laboratory analysis, and analytical data review.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

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8.0 DEFINITIONS

Drum - Any container used to store hazardous materials in a quantity less than 60 U.S. gallons.

Hazardous Materials - Any substance capable of producing deleterious health effects, upon any form of skin contact, inhalation or ingestion by animals or humans.

Investigation-Derived Waste (IDW) - IDW is a subset of remediation wastes. IDW is waste that is generated in the process of investigating or examining an actual or potentially contaminated site. It includes solid and hazardous waste, media (including groundwater, surface water, soils, and sediments) and debris that contain *listed* hazardous wastes or exhibit a characteristic of a hazardous waste. It includes media and debris that is not hazardous but is contaminated with hazardous constituents. Not all IDW is hazardous waste.

9.0 ATTACHMENTS

Attachment 1 – Example Non-Hazardous Waste Label

Attachment 2 – Example Hazardous Waste Label

Attachment 1
Example Non-Hazardous Waste Label

Attachment 2
Example Hazardous Waste Label

Earth Resources Technology, Inc. Standard Operating Procedure **LOW-FLOW GROUNDWATER SAMPLING**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-06-5A**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
Signature	1/23/09 Date	Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to obtain groundwater samples that are representative of the source from which they are taken and minimize the sampler's exposure to groundwater contaminants using low flow purging techniques. The methods and equipment described are for the collection of groundwater samples from the saturated zone of the substrata.

2.0 SCOPE

This procedure provides information on proper equipment and techniques for low flow ground water sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described should be followed whenever applicable, noting that site-specific conditions and project-specific work plans, may require adjustments in methodology.

3.0 REQUIREMENTS

Generally, sampling can be initiated upon parameter stabilization during low flow purging. If an in-line device is used to monitor water quality parameters, it should be disconnected or bypassed during sample collection. The sampling flow rate may remain at the established low flow purge rate or may be adjusted slightly to minimize aeration, bubble formation, turbulent filling of sample bottles or loss of volatiles due to extended residence time in the tubing.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that the procedures described in the project-specific Sampling and Analysis Plan (SAP), at a minimum meet the requirements set forth in this procedure and those detailed in applicable federal and state guidance documents, and that the field team conducting the sampling are adequately trained and briefed on the prescribed methodology.

4.2 Team Leader

The Field Manager is responsible for ensuring that groundwater sampling is conducted in accordance with the procedures outlined in this procedure and in the project-specific SAP. At a minimum, this may include reviewing the SAP for project specific requirements, ensuring that the sampling team is briefed and trained on the project specific requirements, and overseeing the collection and management of the environmental samples.

4.3 Field Team

The Field Team is required to adhere to the sample collection requirements described in this procedure and the project-specific SAP. Deviations from these documents must first be consulted with the Field Team Leader and Project Manager. The Field Team is required to

properly manage the sample collection and handling process of samples from the point of sample collection to relinquishment of samples to the analytical laboratory or designated courier.

5.0 EQUIPMENT

Sample containers will conform to EPA regulations for cleanliness and analysis specific preservation. Ideally, sample withdrawal equipment should be completely inert, economical, easily decontaminated or dedicated disposable able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

The following pieces of equipment may be needed to collect groundwater samples:

- Sample Containers
- Nitrile Gloves
- Coolers for Sample Shipping and Cooling
- Ice to keep samples under 4º Celcius (No blue ice or other forms of ice packs should be used)
- Zip-loc type reclose able plastic bags for sample segregation and wet ice containment
- Appropriate Packing Cartons, Filler, and cushioning material
- Labels
- Clear Packing Tape
- Chain-of-Custody Documents
- Stopwatch
- Graduated Cylinder
- Multi parameter meter.
- Flow through cell with fitting adapters, typically a Horiba U-22 with flow through cell
- Photoionization Detector (PID), or similar instrument with calibration gas
- Appropriate Keys (for Locked Wells)
- Tape Measure
- Water-Level Indicator
- Appropriate Sampling Gloves (Nitrile)
- Purge data forms
- Multi-tool
- Folding table
- Plastic Trash Bags (contractor grade/heavy mil)
- Polyethylene sheeting
- Indelible Marking Pens
- Black, Permanent Ink Pens
- Submersible Pump with controller

  Sample specific tubing such as Teflon®, Polyethylene and Polypropylene (tubing type shall be selected based on specific site requirements and must be chemically inert to groundwater being sampled)

- Teflon®-Coated Wire, Stainless Steel Single Strand Wire, Polypropylene Monofilament Line, or One-Quarter Inch Nylon Rope and Tripod-Pulley Assemble (if necessary)
- Minimum of three 5 gallon buckets
- Decontamination material (distilled water, deionized, analyte free water, Alconox or Liquinox, isopropanol, where applicable)
- Multi-stage decontamination station

6.0 PROCEDURE

To be useful and accurate, a groundwater sample must be representative of the particular saturated zone of the substrata being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis in order to minimize any changes in water chemistry.

Sampling of multiple wells at a single site will always begin with the suspected least impacted well and progress sequentially to the suspected most impacted well, if known, unless project plans specify otherwise. Generally, volatile (e.g., solvents and fuel constituents) and gas sensitive (e.g., containing iron, methane, sulfides) parameters will be sampled first. If an in-line device is used to monitor water quality parameters, it must be disconnected or bypassed during sample collection. Field filtering of samples may be required for certain types of samples. If so, these samples volumes will be collected last.

Methods for low flow purging of monitoring wells prior to sampling include the use of low-flow pumps capable of maintaining flow rates of between 0.1 L/min and 1 L/Min. Procedures described in the following section will be used for purging wells prior to sampling. All water purged prior to sampling will be managed, containerized or treated and discharged, as indicated in the project-specific work plan.

The primary limitations to the collection of representative groundwater samples including, mixing of stagnant water within the monitoring well and representative groundwater from the local aquifer; disturbance and resuspension of settled solids within the monitorign well or in close proximity to the screen; introduction of atmospheric gases or degassing during sample handling and transfer, or inappropriate use of a vacuum sampling device.

6.1 Sampling Approach

General low-flow sampling techniques will consist of the following procedures and be consistent with the project-specific SAP.

6.2 Sampling Methods

The collection of a groundwater sample is made up of the following sequential steps:

6.2.1 Monitoring Well Access

Access the monitoring well head and screen with PID for volatile compounds. When practical, clean plastic sheeting should be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface. A folding/portable table can aid in the management of samples and equipment during the purging process.

6.2.2 Measure Groundwater Level

Measure the static groundwater level in the monitoring well per ERT SOP-07-2 with a water-level indicator and/or interface probe. Calculate the groundwater volume within the well using the acquired data and well construction details.

6.2.3 Install Submersible Pump

Lower the submersible pump, safety cable, tubing and electrical lines slowly (in order to minimize disturbance) into the water column to the approximate midpoint of the groundwater zone to be sampled. Refer to the Sampling and Analysis Plan or ERT Sampling Work Order for specific sampling depth. In cases where this information is not available, the field sampler will provide criteria used for selecting the sampling depth and keep this record in the field logbook. Where possible the pump intake will be staged at least two feet above the bottom of the monitoring well, in order to minimize disturbance of particulates present within the well. Collection of turbid free water samples may be especially difficult if there is two feet or less of standing water in the well. Connect tubing attached to the submerged pump to the influent on the flow-through cell. Secure additional tubing to the effluent plug on the flow-through cell and allow for enough slack so that the effluent tubing can reach the dedicated purge water container.

6.2.4 Measure Groundwater Level

Before starting pump, the groundwater level will be measure once again and recorded in the field logbook. If a data recording pressure transducer is being utilized, initialize the starting condition.

6.2.5 Purge Well

All wells will be purged of stagnant water prior to sampling to ensure that the collected groundwater sample is representative of the target groundwater aquifer.

6.2.5.1 Initial Low Stress Sampling Event

Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Gauge any associated change in the groundwater level. Adjust pump speed accordingly until there is little or no groundwater level drawdown (typically less than 0.25 feet). If the minimal drawdown that can be achieved exceeds 0.25 feet but remains stable, continue purging until groundwater quality field parameters have stabilized. Monitor and record the groundwater level and pumping rate every three to five minutes during purging. Record any pumping rate adjustments (both time and flow rate) made while purging. Pumping rates should be reduced to the minimum required to minimize drawdown and to stabilize groundwater quality indicator parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" as pump flow adjustments are made. Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic), and/or the use of dedicated equipment. If the recharge rate of the well is less than the lowest extraction rate capabilities of the submersible pump and the well is essentially dewatered during purging, then the well should be sampled as soon as the water level has recharged sufficiently to collect the appropriate volume needed for all sample analysis. Regardless if the groundwater quality indicators have stabilized, monitoring wells which have been completely dewatered during purging may be sampled as soon as the groundwater recharges to a volume adequate to collect the appropriate sample volume. Ideally the intake should not be moved during this recovery period. If circumstances do not allow for this, equipment may be removed and the well

may be sampled within a 24 hour period without re-purging the well. Samples may then be collected even though the indicator field parameters have not stabilized.

6.2.5.2 Subsequent Low Stress Sampling Events

After synoptic water level measurement round, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practical, the intake depth and final pump extraction rate from previous sample event(s). Perform purging operations as above.

6.2.6 Monitor Indicator Field Parameters

During well purging, groundwater quality indicator parameters should be monitored using the Horiba U-22 with flow through cell, or similar, every three to five minutes or as specified in the Sample and Analysis Plan. During the early phase of purging, groundwater quality indicators may be very erratic. Purging is considered complete and sampling may commence when all pH, specific conductivity, dissolved oxygen, and oxygen reduction potential have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

Parameter	Stabilization Criteria
pH	± 0.2 unit
Specific Conductivity	± 3%
Dissolved Oxygen	± 10% or 0.2 mg/l whichever is greater
Oxidation Reduction Potential	± 20 mv

Note: The field measurement of temperature and conductivity are not reliable indicators for differentiating between "stagnant" and native formation water due to the fact that each can be influenced by the operation of the mechanical pump. They will not be utilized as stabilization parameters; however their measurements will be recorded.

All recorded groundwater quality indicator data will be acquired using a Horiba U-22 with flow through cell. Transparent flow-through cells are preferred as they allow field personnel to monitoring the accumulation of particulates, free product or other foreign objects within the cell. Accumulation of such constituents within the cell may affect measurements of groundwater quality indicator field parameter values. If the requires cleaning during purging operations, purging may continue. Disconnect the influent tubing from the flow through cell over the container dedicated for purge water and arrange so that the purge water will discharge directly into the dedicated container. After cleaning, the flow-through cell may be reintroduced to the purge line. In order to ensure accurate data, monitoring probes within the flow through cell must be continuously submerged in purge water.

6.2.7 Collect Water Samples

Water samples for laboratory analyses must be collected before water has passed through the flow-through cell. In order to do so, connect a by-pass assembly or disconnect the influent tube from the flow-through cell. Volatile organic compound (VOC) samples will be collected first and placed directly into laboratory certified volatile organic analysis (VOA) sample containers. All sample containers will be filled by allowing the groundwater from the discharge tubing to flow gently down the inside of the sample container with minimal turbulence. During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that 1/4 inch or 3/8 inch

(inside diameter) tubing be used to help insure that the sample tubing remains water filled. Preserve filtered water sample immediately. Label each sample as collected and cover with clear packing tape in order to maintain the integrity of the label. At a minimum, all sample labels will include the sample ID, sample time and date, the samplers ID and the requested analysis. Secure samples per ERT SOP-06-7.

Note: filtered groundwater samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health risk calculations.

6.2.8 Post Sampling Activities

After collection of the samples, in some cases the pump tubing may be dedicated to the well for resampling (by securely hanging the tubing inside the well), decontaminated, or properly discarded. If sample tubing is to be dedicated to the monitoring well, it must be secured in such a fashion as to not allow for the tubing to fall into the well. Every attempt will be made to clear objects from the monitoring well if inadvertently introduced to the well and noted in the appropriate field logbook.

Secure the well head and protective cover.

6.3 Collection of Split Samples or Field Duplicates

Whenever field duplicates are collected or samples are split with another organization the additional samples for identical analyses will be collected along with the original sample (i.e., containers for all volatile organic analyses will be filled first and together, all semi-volatiles together and in proper sequence, and so forth until all sample parameters are in the proper containers).

6.4 Sample Containers

For most samples and analytical parameters, either glass or plastic containers are satisfactory. ERT SOP-06-7, Packaging and Shipment of Field Samples, describes the required sampling containers and shipment methods for various analytes and media.

6.5 Preservation of Samples and Sample Volume Requirements

Sample preservation techniques and volume requirements depend on the type and concentration of constituent and type of analysis to be performed. The project-specific Sample and Analysis Plan will detail sample preservation and volume requirements constituents to be sampled for during the field activities.

6.6 Field Filtration

Field collection and preparation of groundwater samples may include some form of filtration. Field filtration must occur immediately upon or during collection. The recommended method of filtration is via the use of a disposable in-line filtration module (2.0 micron filter for metals and 0.30 micron filter for gross alpha/gross beta). Pressure provided by the dedicated pump will be used to force groundwater through the in-line filter and into the sample container. Prior to using the in-line filter, the filter should be pre-rinse distilled water. Samples for organic analyses should never be filtered. Refer to ERT SOP 06-8 for detailed procedures for field diltration of groundwater samples.

6.7 Handling and Transporting Samples

After collection, samples should be handled as minimally as possible. Samples will be staged in an controlled environment not to exceed 4°C. This can be achieved by placing the samples in a

dedicated refrigerator or cooler with zip-lock bags filled with natural ice. Steps should be taken to ensure that ice melt does not cause sample containers to be submerged and thus introduce the possibility of cross-contaminated or affecting the sample label integrity. All sample containers will be enclosed in dedicated plastic bags to prevent cross-contamination. Sample packing and transportation requirements are described in ERT SOP-06-7.

6.8 Sample Holding Times

Holding times, allowed time between sample collection and analysis for routine samples, are provided in the project-specific Sample and Analysis Plan. This information should be reviewed by the Field Team prior to the start of sampling activities to ensure sample analysis occurs within the allotted timeframe.

6.9 Records

Records will be maintained for each sample that is taken within the dedicated field logbook. The record will include the sample location, total well depth, depth to groundwater, observed groundwater quality indicators, pumping rates, data and time of sample collection and any other applicable data.

6.10 Chain-of-Custody

Chain-of-custody procedures are necessary for adequate sample handling. ERT SOP-06-7 details the requirements for appropriate completion of a chain-of-custody.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

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Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York.

8.0 DEFINITIONS

Equipment/Rinse/Rinsate Blanks - A sample that is collected by pouring over or running analyte-free water through the sample collection equipment after decontamination and before sample collection. The sample is collected in the appropriate sample container with the proper preservative, identical to the samples. This represents background contamination resulting from the field equipment, sampling procedure, sample container, preservative, and shipment.

Environmental Sample - Low concentration sample typically collected offsite and not requiring DOT hazardous waste labeling as a high hazard sample.

Field Blank - In the field, analyte-free water is collected into a sample container with preservatives. The sample containers are the same lot used for the environmental samples. This evaluates contamination introduced from the sample container(s) with applicable preservatives. Field blanks are not used for volatile samples.

Field Replicates/Duplicates - Two or more samples collected at the same sampling location. Field replicates should be samples collected side by side or by collecting one sample and immediately collecting the second sample. Field replicates represent the precision of the whole method, site heterogeneity, field sampling and the laboratory analysis.

Field Split Samples - Two or more representative subsamples taken from one environmental sample in the field. Prior to splitting, the environmental sample is homogenized to correct for sample heterogeneity that would adversely impact data comparability. Field split samples are usually analyzed by different laboratories (inter-laboratory comparison) or by the same laboratory (intra-laboratory comparison). Field splits are used to assess sample handling procedures from field to laboratory and laboratory's comparability.

Filter Blank - In the field, analyze-free water is passed through a filter and collected into in the appropriate sample container. The filter blank is then preserved. This procedure is identical to the sample collection.

Hazardous Waste Sample - Medium to high concentration sample (e.g., source material, sludge leachate) requiring DOT labeling and Contract Lab handling as a high hazard sample.

Laboratory Quality Samples - Additional samples will be collected for the laboratory's quality control: matrix spike, matrix spike duplicate, laboratory duplicates, etc.

Shipping Container Temperature Blank - A water sample provided by the laboratory that is transported to the laboratory to measure the temperature of the samples in the cooler.

9.0 ATTACHMENTS

Low-Flow Groundwater Sampling Form

LOW FLOW SAMPLING DATA SHEET

SITE: _____ FIELD PERSONNEL: _____

DATE: _____ _____

WEATHER:

Earth Resources Technology

WELL DEPTH: _____ STICK-UP / FLUSH MOUNT

MONITORING WELL: _____ WELL SIZE: _____ Inches WELL VOLUME: _____ GAL

2 inch = 0.163 gal/ft 4 inch = 0.653 gal/ft

PID/FID READINGS BACKGROUND: _____ PUMP INTAKE DEPTH: _____ ft below TOC

BENEATH WELL CAP: DEPTH OF WATER BEFORE PUMP INSTALLATION: ft below TOC

TIME	PURGING	SAMPLING	pH (pH units)		SPECIFIC CONDUCTIVITY (mS/cm)		TURBIDITY (NTU)		DISSOLVED OXYGEN (mg/l)		TEMPERATURE (degrees C)		OXYGEN REDUX POTENTIAL (mv)		PUMPING RATE (ml/min)	DEPTH TO WATER (ft below toc)
			READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE	READING	CHANGE		
				NA		NA		NA		NA		NA		NA		
Stabilization			± 0.2		± 3%		± 10% or <10 NTU		± 10% or 0.2 mg/L		± 3%		± 20 mV			

COMMENTS: GAL PURGED:

Earth Resources Technology, Inc. Standard Operating Procedure **PACKAGING AND SHIPMENT OF FIELD SAMPLES**			
Effective Date: **01/27/09**	Version: **001**	SOP#: **ERT SOP-06-7**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/28/09 Date	 Signature	1/27/09 Date

1.0 PURPOSE

The purpose of this procedure is to define the requirements necessary for sample packaging and information to be included on sample labels and chain-of-custody (COC) records used in sample transfer from field personnel to the analytical laboratory.

2.0 SCOPE

This procedure applies to the packaging, shipping and documentation of samples being collected during field activities and transferred contracted laboratory for analysis. Specifically, this document outlines shipping and sample documentation procedures in accordance with the U.S. Department of Transportation (DOT) guidance and regulation. This procedure is applicable to all environmental samples collected for delivery to analytical laboratories; however, this procedure does not take precedence over federal, state or project-specific requirements for sample management and delivery to the analytical laboratory.

3.0 REQUIREMENTS

Careful packaging, shipping and documentation are essential to insure that collected samples are received undamaged and authenticated by the contract analytical laboratory.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for providing project-specific guidelines within the Sampling and Analysis Plan (SAP) for the proper handling and management of environmental samples, and ensuring that the field team is properly trained in the appropriate procedures. Upon completion of the sample accusation and COC completion, the Project Manager is responsible for reviewing the collected samples and the completed COC for accuracy.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that samples are properly packaged, labeled, documented on the COC and shipped to the contracted analytical laboratory in accordance with this procedure and within the project-specific SAP. The Field Manager will also ensure that proper sample management techniques are followed for the entire duration that samples are in the custody of ERT personnel.

4.3 Field Team

The Field Team is responsible for implementing packaging, labeling, documenting and shipping requirements detailed in this procedure and the project-specific SAP.

5.0 EQUIPMENT

- Cooler
- Packing Tape
- Bubble Pack
- Sampling Gloves
- Heavy Duty Plastic Trash Bags
- Inert absorbent material/matting
- COC
- Pen, Black Ink
- Ziploc-type Plastic Bags
- Permanent Felt Tip Marker
- Shipping documents
- Right-side-up and Fragile Placarding
- Custody Seals
- Natural Ice
- Temperature Blanks

6.0 PROCEDURE

6.1 Sample Packaging and Shipping

Typically, samples collected for shipment to analytical laboratories will classified as non-hazardous environmental samples. ERT will not ship any material known to be hazardous. If hazardous material is suspected, the Project Manger will be notified and appropriate accommodation will be made to transfer the sample media from the site. If hazardous material is expected to be encountered during filed activities, the project-specific SAP will details the proper procedures for handling the hazardous media.

6.2 Packaging

Environmental samples will be packaged following the procedure outlined below:

- Conduct a “bottle count” to ensure correct sample volumes for the analytical procedures requested, correct number of samples, and that information on each sample label matches the COC and complies with the project-specific SAP.
- Line shipping cooler with plastic trash bag
- Line bottom of cooler with inert absorbent material/matting.
- Tape drain plug closed (if applicable).
- Place doubled-bagged wet ice on the bottom of the lined shipping cooler. Enclose temperature blank in bagged ice, sufficiently protected from breakage.
- Place inert cushioning material on top of ice.
- Check to ensure that sample lids are tightened to prevent leakage and with enough headspace (except VOC containers with a septum lid) to compensate for any pressure and temperature changes during shipment (approximately 10 percent of the volume of the container).
- Segregate samples from sample points into separate Ziploc-type plastic bags to minimize the potential for cross contamination and contact with any liquid generated from melting ice.
- Arrange samples within the cooler as to minimize contact between breakable sample containers.
- Fill any void spaces between samples with inert cushioning material.
- Place double-bagged ice on top of the samples.
- Close plastic bag lining cooler and tape shut.

- Place signed Chain-of-Custody form into Ziploc-type plastic bag and affix to inside of lid of the shipping cooler with packing tape. In the instances of multiple coolers for a single Chain-of-Custody place photo copies of original COC in Ziploc-type bag in each additional cooler.
- Tape the lid of the shipping cooler shut including around the edge where the lid contacts the body.
- Place COC seals across the threshold where the cooler lid contact the body of the shipping cooler, or as detailed in the project specific SAP.
- Tape down any movable external handles.
- Affix shipping documents to the lid.
- When multiple coolers are shipped during a single event they should be numbered sequentially on the exterior (1 of 3 etc.) so that the receiving facility would be aware of any irregularities in shipment.
- "THIS SIDE UP" or "THIS END UP" and "FRAGILE" placards and upward-pointing arrows should also be affixed on the outside of the cooler.

6.2.1 Shipping Papers

No DOT shipping papers are required for the transport of non-hazardous environmental samples. However, appropriate COC forms must be included with the shipment.

6.2.2 Transportation

There are no DOT restrictions on mode of transportation for non-hazardous environmental samples.

6.3 Chain-of-Custody Guidelines

A properly completed COC ensures that handling and shipment of environmental samples has been conducted in a defensible and scientific manner. COC procedures track environmental samples from the time and place it is first obtained to the analytical laboratory. These procedures also provide an auditable trail of evidence the samples pass from the custody of one individual to another. In addition, procedures for consistent and detailed records facilitate the admission of evidence under Rule 803(b) of the Federal Rules of Evidence (P.L. 93-575).

COC procedures, record keeping, and documentation are an important in ensuring the quality of sample analytical results. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

6.3.1 Sample Identification

The following information shall be recorded in the Field Logbook when samples for laboratory analysis are collected:

- unique sample number
- source of sample (including name, location, and sample type)
- time of collection
- number of samples collected
- types of sample container
- preservative used
- analysis required

- field observations (include pH, temp, depth to water) when applicable
- equipment used to make physical measurements and collect samples
- calibration data for equipment used

6.3.2 Sample Labels

Samples, other than in-situ measurements, are removed and transported from the sample location to a contracted analytical laboratory or other location for analysis. Before transport, however, the collected sample volume is often divided into various containers for particular analytical procedures. Each subsample will be containerized and preserved in accordance with the project-specific SAP.

Each sample container will have a dedicated sample label identifying the sample. The following table describes the minimum sample label requirements.

Minimum Sample Label Requirements	
Field Sample No.	The unique sample number identifying this sample as prescribed by the Work Plan, SAP and/or QAPP
Project Name	Name
Project No.	ERT Project number
Date	A six-digit number indicating the month, day, and year of sample collection; e.g., 12/21/85
Time	A four digit number indication the 24-hour time of collection (for example: 0954 of9:54 a.m., and 1625 is 4:25 p.m.)
Media Type	Water, Soil, etc.
Method Type	Grab or Composite
Analysis	Method of analysis for the laboratory
Preservation	Type of preservative
Collector's Initials	Initials of the sampler

Once the required information is entered onto the label and affixed to the sample container, clear packing tape should be placed over the label to maintain the integrity of the label.

6.3.3 Chain-of-Custody Procedures

After collection, separation, and identification, the sample is maintained under COC procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

6.3.4 Field Custody Procedures

1. Samples are collected as described in the project-specific SAP. Care must be taken to ensure that the sample identification on the label exactly matches sample identification on the COC.

2. Field personnel collecting the environmental samples are responsible for the care and custody of the samples collected until they are properly transferred to a predetermined staging area, the custody of another Field Team member, or the custody of a currier.

3. Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions.

6.3.5 Transfer of Custody and Shipment

Samples are accompanied by a COC Record Form. When transferring the possession of samples, both the individual relinquishing and receiving the samples will sign, date, and note the time of transfer on the COC. The COC documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory.

Typically the COC is filled out in its entirety as follows:

1. Enter project specific information (i.e. project number and name, Contract Lab case No. or SAS No.). For each sample, record the applicable Sample Identification, date and time of sample collection, the sampler, preservation information, type of sample (composite/grab), number of containers transferred and the requested analytical parameters.
2. Sign, date, and enter the time under "Relinquished by" entry.
3. Ensure the person receiving the sample signs for the "Received by" entry, or enter the name of the carrier (e.g., UPS, Federal Express) under "Received by".
4. Record the bill-of-lading or Federal Express air bill number under "Remarks or Reason for Change of Custody", if appropriate.
5. Place the original (top, signed copy) of the Chain-of-Custody Recorded Form in the appropriate sample shipping package. Retain a copy with field records.

The custody record is completed using black waterproof ink. Any corrections are made by drawing a single line through error and initialing the change, then entering the correct information. Erasures are not permitted.

Common carriers (FedEx and UPS) will not accept responsibility for handling COC Forms, necessitating the package of the COC Record in the sample container (enclosed with the other documentation in a plastic zip-lock bag secured to the lid of the shipping cooler). As long as custody forms are secured inside the sample shipping container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the COC, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

7.0 REFERENCES

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 1992. *RCRA Ground Water Monitoring: Draft Technical Guidance.* November

United States Environmental Protection Agency (USEPA), 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, Region 4. November.

United States Environmental Protection Agency (USEPA), 2007. *Field Branches Quality System and Technical Procedures*. Region 4 Science and Ecosystem Support Division. SESDPROC-209-R1. November.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

Carrier - A person or firm engaged in the transportation of passengers of property.

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to the subsequent custodian. Attachment 1 shows a typical Chain-of-Custody Form. Chain-of-Custody Form is a controlled document. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under your custody if:

- You possess the sample.
- It is in your view, after being in your physical possession.
- It was in your physical possession and then you locked it up to prevent tampering.
- You have designated and identified a secure area to store the sample

Environmental Sample - A low concentration sample typically collected offsite and not requiring DOT hazardous waste labelling as a high hazard sample.

Marking - Applying the descriptive name, instructions, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 172, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank car tanks.

Placard - Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all four sides of a vehicle transporting certain hazardous materials.

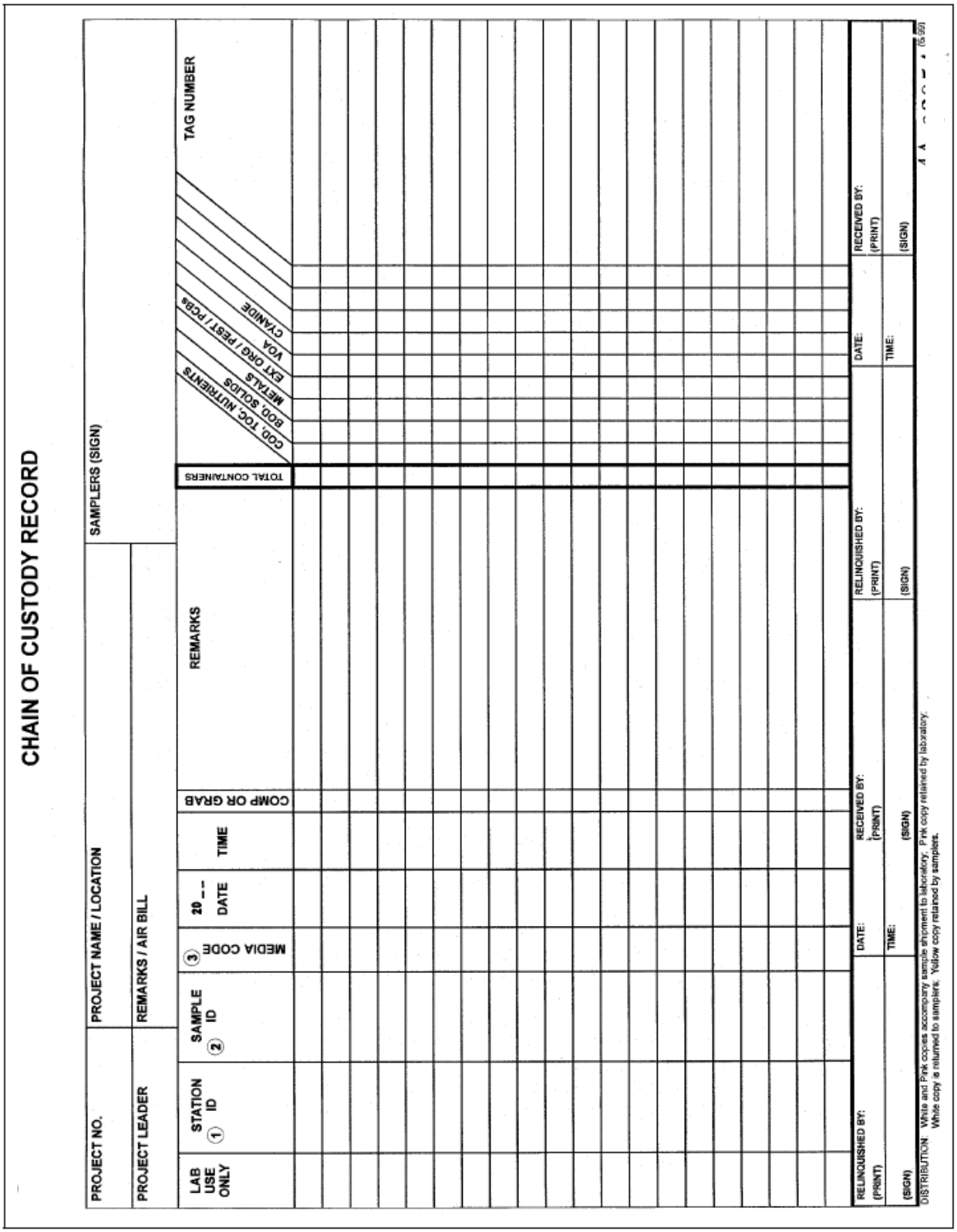
Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

9.0 ATTACHMENTS

1 – Example Chain of Custody Form

2 – Example Custody Seal

Attachment 1-Example Chain of Custody Form



CHAIN OF CUSTODY RECORD

PROJECT NO. | PROJECT NAME / LOCATION | SAMPLERS (SIGN)

PROJECT LEADER | REMARKS / AIR BILL

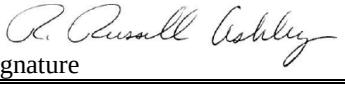
LAB USE ONLY	STATION ID ①	SAMPLE ID ②	MEDIA CODE ③	20__ DATE	TIME	COMP OR GRAB	REMARKS	TOTAL CONTAINERS	COD, TOC, NUTRIENTS	BOD, SOLIDS	METALS	EXT ORG / PEST / PCBs	VOA	CYANIDE	TAG NUMBER

RELINQUISHED BY: (PRINT)	DATE:	RECEIVED BY: (PRINT)	RELINQUISHED BY: (PRINT)	DATE:	RECEIVED BY: (PRINT)
(SIGN)	TIME:	(SIGN)	(SIGN)	TIME:	(SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory. Pink copy retained by laboratory.
White copy is returned to samplers. Yellow copy retained by samplers.

Attachment 2 – Example Custody Seal

Example Custody Seal

Earth Resources Technology, Inc. Standard Operating Procedure **FIELD FILTRATION OF WATER SAMPLES**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-06-8**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/23/09 Date	 Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to establish uniform guidelines for filtering water samples in the field.

2.0 SCOPE

This procedure applies to the field filtration of water samples. Groundwater sampling procedures are outlined in ERT SOP-06-5. Surface water sampling procedures are outlined in ERT SOP-06-2.

3.0 REQUIREMENTS

The field or laboratory filtration of water samples is required for several analytical procedures including analysis for dissolved metals. Two field filtration methods are described in this procedure, in-line positive pressure filtration and negative pressure filtration. Positive pressure filtration is the preferred methodology; however negative pressure filtration may be required, particularly for surface water samples.

Samples collected for organic analysis should never be field or laboratory filtered.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager will be responsible for ensuring that field personnel have been trained in the use of this procedure and the methodology is consistent with the Sampling and Analysis Plan (SAP).

4.2 Field Team Leader

The Field Team Leader will be responsible for ensuring filtration procedures associated with water sample collection comply with those described in this procedure and project-specific SAP.

4.3 Field Team

The Field Team will be responsible for compliance with this procedure and the project-specific SAP.

5.0 EQUIPMENT

The following equipment may be used for the filtration of water samples:

- Prepackaged Disposable In-Line Filters (sizing applicable to project specific and analysis specific constraints).
- High Density Polyethylene (HDPE) or Teflon® Tubing
- Nalgene filter funnel/collection flask

- Sampling Pump
- Hand-operated vacuum/pressure pump
- Sample Bottles

6.0 PROCEDURE

6.1 Positive Pressure Filtration

1. Use High Density Polyethylene (HDPE) or Teflon® tubing for pump and filter connections.
2. Connect the appropriately sized in-line filter to the discharge tubing from the pump. Make sure the flow arrow on the filter is pointing in the correct direction and the system is leak-proof.
3. Apply pressure to the liquid sample (via pump) to force it through the filter.
4. Allow 500-1000 ml to flow through the inline filter to allow equilibration of the filter media (manufactures recommendations should also be consulted)
5. Position discharge of filter directly into the appropriate sample container.
6. Replace the in-line filter when the flow becomes too restricted because of a buildup on the filter. To replace the filter, discontinue pumping (turn off pump), relieve the pressure in the system (line between the pump and the filter), and disconnect the filter and replace with a new one.
7. Verify that a Teflon® liner is present in the cap. Secure the cap tightly.
8. Label the sample bottle with an appropriate label. Be sure to include all necessary information.
9. Record the information in the field logbook and field sheet, and complete all chain-of-custody documents (refer to ERT SOP-06-7).
10. Release the pressure in the filtration equipment, disconnect sample filtration device from the discharge tubing, and thoroughly decontaminate or properly dispose of all equipment and materials in accordance with the project sampling and analysis plan.

6.2 Negative Pressure Filtration

1. Select a pre-sterilized filter assembly with a filter of appropriate pore size.
2. Connect vacuum tubing to the pump and the filter assembly. Use High Density Prolyethylene (HDPE) or Teflon® tubing for pump and filter connections and verify that it is leak-proof.
3. Pour the aqueous sample into the filter funnel portion of the filtration assembly. Avoid excessive turbulence or agitation of the sample, or transferring solids that may have settled to the bottom of the sample container.
4. Using a vacuum pump, create a negative pressure as recommended by the equipment manufacturer in the collection vessel of the filtration assembly to start the filtration process.
5. Collect the filtrate (sample) into the collection flask or other vessel.
6. Replace the filter funnel portion of the assembly when the filter becomes too restricted because of solids buildup on the filter. To replace the filter, depress the pressure/vacuum release button, disconnect the filter funnel and replace it with a new one, create a vacuum with the hand pump, and continue filtering the remaining sample.

7. Release the negative pressure at the vacuum pump and in the filtration equipment; disconnect the collection flask.
8. Transfer the filtrate from the collection flask into appropriate sample containers, avoiding excessive turbulence or agitation to the sample.
9. Verify that a Teflon® liner is present in the cap. Secure the cap tightly.
10. Label the sample bottle with an appropriate label and Sample ID. Be sure to include all necessary information.
11. Record the information in the field logbook and/or field sheet, and complete all chain-of-custody documents (refer to ERT SOP-06-7).
12. Discard or decontaminate all sample filtration equipment and materials in accordance with the project-specific Sampling and Analysis Plan.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 1989. *Ground Water Sampling for Metals Analysis.* Superfund Ground Water Issue, EPA/540/4-89/001. March.

United States Environmental Protection Agency (USEPA), 1996. *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedure.* Ground Water Issue, EPA/540/S-95/504. April.

United States Environmental Protection Agency (USEPA), 2002. *Ground-Wager Sampling Guidelines for Superfund and RCRA Project Managers*. Ground Water Forum Issue Paper, EPA/542/S-02/001. May.

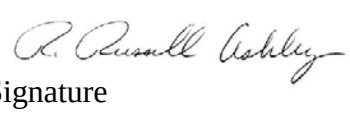
Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

None

9.0 ATTACHMENTS

None

Earth Resources Technology, Inc. Standard Operating Procedure **EXPLOSIVES FIELD SCREENING**		
Effective Date: **03/25/2009**	Version: **001**	SOP#: **ERT SOP-06-10**
Approvals		
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager
 Signature	4/28/09 Date	 Signature	4/28/09 Date

1.0 PURPOSE

The purpose of this procedure is to define the requirements for performing the field screening for explosive compounds using commercially available text kits (Expray® or DropEx®). Both kits can be used for the detection of nitroaromatics, nitrate esters and nitroamines, and inorganic nitrate-based explosive compounds. The difference between the two test kits is one is aerosol-based (Expray®) and the other uses a dropper (DropEx®). Both require the use of collection paper (which is provided in each kit).

2.0 SCOPE

Expray®/DropEx® test kits can be used to perform field screening for explosives (in soil or on surfaces) to aid in determining sample locations for analytical analysis. Both test kits use colorimetric reactions to screen samples and can provide an order of magnitude estimate of the level of explosive compounds in the sample. Both kits follow the same basic process to determine the different types/groups of explosives within the sample. Shipping, shelf life, and sensitivity are the key factors to consider when making a determination of which test kit to use. The Expray® kits have a shelf life of roughly 12 months, have a higher sensitivity, and must be shipped as a dangerous good. DropEx® also has a shelf life of 12 months, but must be used within 30 days of opening the packaging, is less sensitive, and can be shipped without any special marking or handling requirements.

Both kits can be used to the detection and identification of the following explosive compounds:

- Group A-including trinitrotoluene (TNT), trinitrobenzene (TNB), dinitrotoluene (DNT), Tetryl, and picric acid and its salts.
- Group B-including dynamite, nitroglycerin (NG), nitrocellulose (NC), Cyclotrimethylenetrinitramine (RDX), Cyclotetramethylenetetranitramine (HMX), Pentaerythritoltetranitrate (PETN), Semtex, C4, and smokeless powder.
- Group C-including nitrate-based explosives, such as black powder, flash powder, gun powder, ammonium nitrate-fuel oil (ANFO) and ammonium nitrate.

3.0 REQUIREMENTS

These commercially available kits come with all necessary supplies, including aerosol cans/droppers, application paper, verification pads, detection charts, material safety data sheets (MSDS), and a carrying case. According to the MSDS, no personal protection equipment (i.e., gloves, eye protection, or respirator) is required when using these kits. However, they should be used in areas with adequate ventilation.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that field personnel have been trained in the use of this procedure ensuring consistency in methodology between Field Personnel and compiling the Expray®/DropEx® data for inclusion into a field report.

4.2 Field Team Leader

The Field Manager/Field Team Leader will be responsible for ensuring that complete documentation is available for Expray®/ DropEx® testing and for overall implementation of this procedure. The Field team Leader is responsible for reviewing logs for completeness and accuracy.

4.3 Field Team

The Field Team is responsible for executing the Expray®/DropEx® test and accurately recording (logging) pertinent information.

5.0 EQUIPMENT

Expray®/DropEx® is sold in a kit configuration, which provides three aerosol sprays/droppers, collection papers, and an RDX-impregnated verification pad (verification pad is useful for ensuring that the spray can/dropper still contains active reagents and for demonstrating how a positive reaction will appear), and a carrying case. Expray®/DropEx® kits are available in both regular size (100 tests) and mini-size (50 tests).

6.0 PROCEDURE

Testing is performed in the following manner:

1. Remove a collection paper from the dispenser. The collection paper is laminated on one side and have a protective cover on the other to prevent either pre- or cross contamination
2. Peel the protective layer exposing the sticky surface. The collection papers are covered with a thin layer of glue to ensure good specimen collection. Be sure not to touch and potentially contaminate the exposed adhesive collection surface.
3. Wipe suspected surface (e.g., walls, floors, door handles, objects) with the sticky side of the collection paper. A small amount of suspected material, soil or extract can be placed directly on the collection paper using aseptic technique.
4. Test the paper with a light application of Expray®-1 or a drop of DropEx®-1. Always shake the Expray® reagents first, direct the spray away from the body and apply from a distance of about six inches. Apply DropEx® from a distance of about three inches.

5. Observe for an immediate color change. If a dark brown color appears (similar to the color on the label) this indicates the presence of Group A explosives. Some compounds may give other colors. Refer to the color chart provided with the kit.
6. If after applying the first reagent there is no color change, then test the same spot on the collection paper with Expray®-2/DropEx®-2.
7. Observe for an immediate appearance of a pink color (similar to the color on the label) indicating the presence of Group B explosives. Most plastic explosives, including Semtex, belong to this group. Tetryl belongs to both Groups, so it will change from orange (color change caused by Expray®-1/DropEx®-1) to pink after being tested with Expray-2/DropEx-2.
8. It is especially important that reagent 3 be well shaken immediately prior to use.
9. If there is no color change after using the 1 and 2 reagents, but presence of explosives is still suspected, test the same spot on the collection paper with Expray®-3/DropEx®-3.
10. Observe for an immediate appearance of a pink color (similar to the color on the label) indicating the presence of inorganic nitrates, which could be part of an improvised explosive.
11. If using the DropEx® kit only, continue with step 12. If using Expray®, the test is complete.
12. If other explosives are suspected, repeat steps 1 to 3 (do not use a sample that was tested with other reagents) and apply DropEx®-A to the fresh sample. Observe for an immediate appearance of a dark-blue color indicating chlorates or bromates are present.
13. If there is no color change after using DropEx® A, but presence of explosives is still suspected, test the same spot on the collection paper with DropEx® B. Observe for the immediate appearance of a dark-blue color indicating peroxide based explosives are present.

Refer to the Detection Charts, presented as Attachment 1.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

Medimpex United, Inc., 2007. Expray® *Explosives Detection*, accessed March 25, 2009. (http://www.meditests.com/exexdetfielt.html)

The Mistral Group. 2007. *Explosives Detection Field Test Kits*, accessed March 25, 2009. (http://www.mistralgroup.com/SEC_explosives.asp)

8.0 DEFINITIONS

None

9.0 ATTACHMENTS

1 Expray®/DropEx® Detection Charts

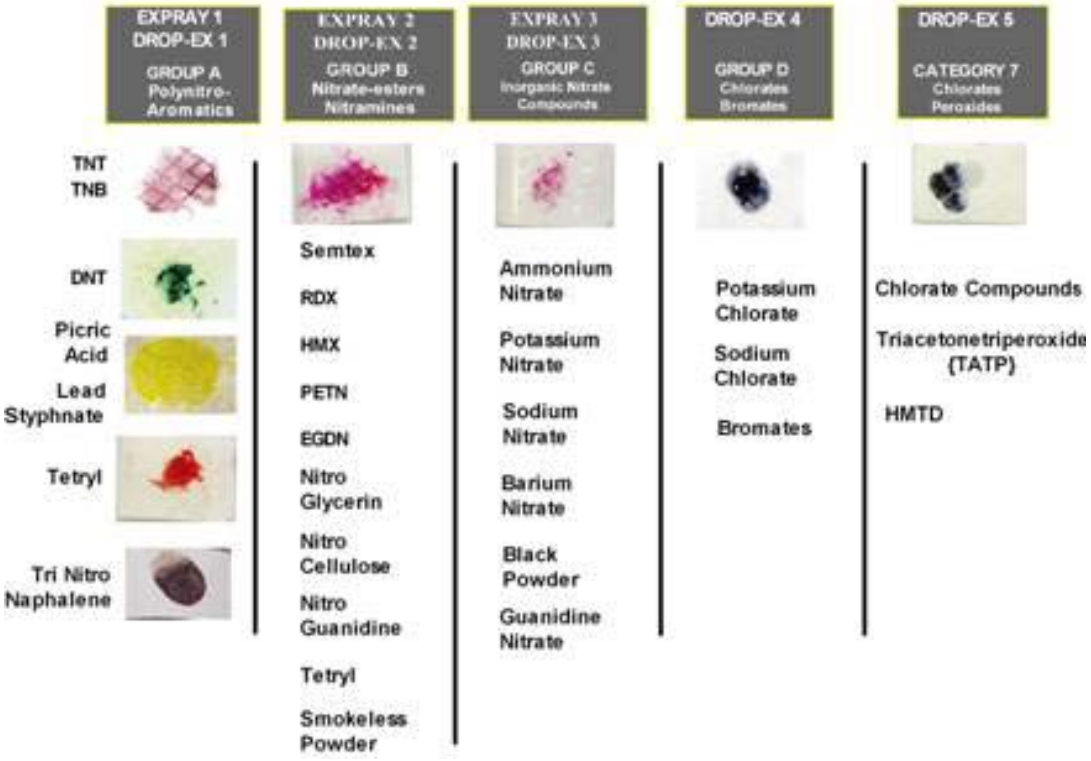
Attachment 1

Expray®/DropEx® Detection Charts

Detection Chart

Product :	EXPRAY - 1 / Drop-Ex 1		EXPRAY - 2 / Drop-Ex 2		EXPRAY - 3 / Drop-Ex 3		Drop-Ex 4 (used alone)	
Detects:	Polynitro Aromatics		Nitrate-esters Nitramines		Inorganic Nitrate Compounds		Chlorates	
	Group A		Group B		Group C		Group D	
#	Substance	Color	Substance	Color	Substance	Color	Substance	Color
1	TNT	Dark Brown	Semtex	Pink	Ammonium Nitrate	Pink	Potassium Chlorate	Dark Blue
2	TNB	Dark Brown	RDX	Pink	Potassium Nitrate	Pink	Sodium Chlorate	Dark Blue
3	DNT	Blue Green	HMX	Pink	Sodium Nitrate	Pink	Bromides	Dark Blue
4	Picric Acid	Yellow	PETN	Pink	Barium Nitrate	Pink		
5	Tri Nitro Napthalene	Violet	EGDN	Pink	BlackPowder	Pink		
6	Lead Styphnate	Yellow	Nitro Glycerin	Pink				
7			Nitro Cellulose	Pink				
8			Tetryl	Pink				
9			Smokeless Powder	Pink				

EXPLOSIVE DETECTION COLOR CHART



Earth Resources Technology, Inc. Standard Operating Procedure **PID OPERATION**			
Effective Date: **3/24/09**	Version: **001**	SOP#: **ERT SOP 06-13**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, P.G. Field QA Manager	
Signature	3/20/09 Date	Signature	3/16/09 Date

1.0 PURPOSE

The purpose of this procedure is to outline the steps necessary for operation and maintenance of photoionization detectors (PIDs) during a field investigation, and should be used in conjunction with the manufacturer's instruction for specific PIDs.

2.0 SCOPE

The PID is a useful general survey instrument at hazardous waste sites, and capable of detecting and measuring real-time concentrations of many organic and inorganic vapors in air. It is similar to a flame ionization detector (FID) in application; however, the PID has somewhat broader capabilities in that it can detect certain inorganic vapors. Though it can be calibrated to a particular compound, the instrument cannot distinguish between detectable compounds in a mixture of gases and, therefore, indicates an integrated response to the mixture.

3.0 REQUIREMENTS

Measurement of organic vapors is an important and necessary requirement during field investigations for the health and safety of workers. Detection of organic vapors during field investigations may also be a precursor of the presence of contaminants at the site. It is important to note that:

- The PID must be calibrated to a specific compound, though it is not a nonspecific total vapor detector. It cannot be used to identify unknown substances; it can only roughly quantify them.
- The PID does not respond to certain low molecular weight hydrocarbons, such as methane and ethane.
- Certain PIDs do not detect a compound if the probe has a lower energy than the compound's ionization potential. Certain toxic gases and vapors, such as carbon tetrachloride and hydrogen cyanide, have high ionization potentials and cannot be detected with a PID.
- Electrical power lines or power transformers may cause interference with instrument measurements.
- High winds and humidity affect instrument measurements.
- If the PID does not function properly, then it should not be used and under no circumstances should work be done that requires air monitoring with a PID.

4.0 RESPONSIBILITIES

4.1 Field Team Leader

The Field Team Leader is responsible for ensuring that the necessary equipment is available for calibration and maintenance. The Field Team Leader should also ensure that the calibration methodology is consistent and that workers have been instructed in the proper use of equipment.

4.2 Field Team

The Field Team is responsible for proper calibration, operation, maintenance, and overall safe handling of the PID according to this SOP and the manufacturer's operating manual.

5.0 EQUIPMENT

- Photoionization Detector (PID), including Attachments (probes)
- Operating Manual
- Calibration Test System, including Calibration Gas
- Battery Charger
- Spare Batteries
- Small Screw Driver
- Teflon tubing for downhole measurements
- Plastic bags for protecting the PID from moisture and dirt

6.0 PROCEDURE

6.1 PID Start-up/Calibration

1. Allow the temperature of the unit to equilibrate to its surrounding. This should take about five minutes, and done in a known "hazard-free" area prior to calibration.
2. Make a zero adjustment to the instrument outside of the work area, by setting the zero potentiometer on the PID to 0.0. Wait 15-20 seconds to ensure zero stabilization.
3. Calibrate the span of the instrument according to manufacturer instructions, by adjusting the instrument reading to the concentration of the span gas solution when connected to the PID. Isobutylene gas is a typical calibration standard (reference operating manual for alternative calibration to a specific compound).

   Note: If the meter reading is greater than ±15% of the response value of the calibration gas used prior to adjustment recalibrate the instrument. If repeated recalibrations does not result in a measurement within ±15% of the response value then the instrument should not be used, and returned to the vendor/manufacturer for service and recalibration.
4. Confirm calibration by screening calibration gas outside of calibration mode (i.e. in measurement mode).
5. Record all instrument and calibration information in the Field Logbook or project-specific field form(s), including manufacture, model number, individual unit serialized ID, type of calibration gas, and gas concentration.

Note: The PID should be calibrated daily at the beginning of the start of field operations and any time that the field personnel feel that the instrument is displaying inaccurate measurements.

6.2 PID Operation (Ambient Screening)

1. Position the probe assembly close to the area to be monitored because the low sampling rate allows for only very localized readings. Under no circumstances should the probe tip assembly be immersed in fluid or exposed to excessive moisture, dirt, or contamination.
2. Monitor the work activity as specified in the site Health and Safety Plan and/or Sampling and Analysis Plan.
3. The PID survey should be conducted at a slow to moderate rate of speed and the intake assembly (the probe) slowly swept from side to side. There is a three to five second delay in read-out depending upon the instruments sensitivity to the contaminant.
4. When the monitoring activity is complete or at the end of the day, check the PID measurement accuracy versus the calibration standard, then carefully clean the outside of the PID with a damp, disposable towel to remove any visible dirt.
5. Record all instrument and field measurement information in the Field Logbook or project-specific field form(s).

6.3 PID Operation (Head Space Screening)

1. Place media to be screened in a new reclosable plastic bag.
2. Allow equilibration, being cognizant of the accumulation of condensation inside the bag.
3. Pierce the bag with a small sharp pointed object or open a small portion of the sealed edge and insert the PID sample tip.
4. Allow sufficient time for vapors entrained in the sample tubing to pass the instrument's sensors.
5. Record all instrument and field measurement information in the Field Logbook or project-specific field form(s).

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

HNU Systems, Inc. 1975. "Instruction Manual for Model PI-101 Photoionization Analyzer."

U.S. Environmental Protection Agency. 1984. "Characterization of Hazardous Waste Sites – A Methods Manual: Volume II, Available Sampling Methods, Second Edition", EPA-600/4-84-076, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, Nevada.

United States Environmental Protection Agency (USEPA), 1994. *Photoionization Detector (PID) HNU.* U.S. EPA Environmental Response Team, SOP 2114. October.

8.0 DEFINITIONS

Photoionization Detector (PID) - a portable, nonspecific, vapor/gas detector employing the principle of photoionization to detect a variety of chemical compounds, both organic and inorganic, in air.

Earth Resources Technology, Inc. Standard Operating Procedure **DRUM HANDLING AND SAMPLING**			
Effective Date: 0**3/24/09**	Version: **001**	SOP#: **ERT SOP-06-14**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, P.G. Field QA Manager	
Signature	3/20/09 Date	Signature	3/18/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide general reference information for use in planning for and implementing sampling programs involving the moving and/or opening of closed containers on uncontrolled hazardous substance sites. Guidelines are provided for selecting containers to be opened and for moving and opening them. In addition, site organization, protective clothing, worker protection and other safety procedures are discussed.

2.0 SCOPE

This guideline is applicable to opening and sampling of closed containers (120 gallon or less) on uncontrolled hazardous substance sites. Bulk tanks such as railroad tank cars, large above-and below-ground tanks (with a capacity of more than 120 gallons), and tank trailers are not considered in this procedure. Bulk tank removal is best handled through the procurement of a subcontractor (See Attachment 1).

3.0 REQUIREMENTS

Strict adherence to safety precautions cannot be overemphasized when handling and sampling drums. Hazards encountered when sampling drums include detonations, fires, explosions, vapor generation and worker exposure to the waste. Scenarios involving drums encountered in the field may include drums that are unmarked, mislabeled, bulging, buried, deteriorated or leaking. Consequently, such drums may require handling to accommodate sampling. Because the condition of the drum and its contents dictate how drums are handled and sampled, no single procedure can be written to cover all possibilities. This procedure lists general guidelines that should be used when developing and on-site drum handling/sampling procedure. The procedure developed should be based on all available information and revised as more information becomes available. When implementing the procedure, common sense and good judgment are paramount.

Consult OSHA regulations (29 CFR Sections 1910 and 1926) for established general requirements and standards for storing, containing, and handling chemicals and containers, and for maintaining equipment used for handling materials.

Consult EPA regulations (49 CFR 265) for requirements pertaining to the types of containers, maintenance of containers and containment structure, and design and maintenance of storage areas.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for determining that opening and sampling of containers is necessary for the field investigation program, and the approximate numbers and types of containers to be opened.

4.2 Field Team Leader

The Field Team Leader is responsible for the overall safe conduct of the container opening and sampling operations. This includes informing and obtaining help from local authorities if necessary; selection of containers to open/sample; testing, moving, and staging of containers; container opening and sampling; resealing; and halting operations, including ordering site evacuation or requesting public evacuation, with help from local authorities, if necessary. The drum opening and sampling program will be planned in detail in the project-specific work plan. If any unexpected results (e.g., explosions, atmospheric releases) occur, the Field Team Leader must inform the Project Manager immediately. Together with the Health and Safety Officer and outside assistance, if necessary, (e.g., EPA's Emergency Response Team), he must determine the most prudent course of action.

4.3 Health and Safety Officer

The Health and Safety Officer is responsible for safety of all on-site operations, alerting the Field Team Leader of any potentially unsafe conditions, and halting work if on-site personnel or off-site public health is threatened.

5.0 EQUIPMENT

- Spill Control Kit
- Drum Overpacks
- Drum Grappler
- Drum Opening Equipment (suitable for the respective type drums)
- Explosion Meter
- Portable Organic Vapor Analyzer
- Fire Extinguisher, Class A, B and C, 12 lb. capacity
- Alpha and Beta Radiation Detector
- Personal Protective Equipment as specified in the project-specific work plan. This may include: Robar or Tingley boots, Tyvek protective suit with acid jacket and pants, vinyl booties, vinyl sample gloves, nitrile outer gloves, hard hat with splash shield, and supplied air respirator.
- Plexiglass Shield
- Drum Sampling Data Sheet
- Chain of Custody Forms
- Appropriate sample
- Drum opening devices
- Absorbent material for spills
- Sheet Plastic
- Sampling Equipment
- Decontamination equipment

- Field Logbook.

6.0 PROCEDURE

6.1 General

The guidance presented is based on field experience in working with containers on uncontrolled hazardous substance sites. It will be evident that in many cases hard-and-fast rules cannot be given, and professional judgment is required because uncontrolled variables are involved. For example, no one can be absolutely certain of any assessment of the potential contents of a container. Labels cannot be absolutely trusted; only educated guesses can be made by a thorough review of all available background data, such as potential sources of the wastes.

Three basic risks are involved in moving and opening closed containers: (1) exposure of personnel to toxic materials, (2) fire, and (3) explosion. The first risk can be reasonably eliminated though the use of proper skin and respiratory protection equipment. The use of level A protection (i.e. totally encapsulated suit in conjunction with a self-contained breathing apparatus) acceptably reduces the risk of a worker being injured by toxic vapors, mists, or splashes. In the same way, standard fire prevention procedures can be used to reduce the fire hazard through the use of detector instruments and proper equipment. These include the use of nonsparking tools and intrinsically safe radios, pumps, and other equipment, as well as the staging of firefighting equipment and the elimination of any other possible ignition sources. The explosive risk, however, is not as easily handled, and thus is the primary consideration in any container-opening operation. Even if no solid evidence of the presence of explosives is found during the preliminary data collection, one can never be certain that explosives have not been disposed of at the site. In order to provide the same reasonable level of protection against this risk as against toxic exposure and fire, a very cautious approach, such as the one recommended in this guideline, should be used.

6.2 Background Review

This section details the elements of a site background review necessary to prepare a Site Operations Plan for drum opening. The decision of whether or not to conduct the operation depends on the assessment of the site history. Therefore, it is important that the following tasks are completed thoroughly.

6.2.1 Preliminary Assessment

The preliminary assessment of existing data should be consulted in planning for a container-opening operation. Of special importance are items that can be used to characterize the types of hazardous material present at the site (e.g., generator records, manifests, inventories, personal interviews, monitoring data). The review of all such data should search for the possible presence of shock-sensitive explosives and/or reactive chemicals. The absence of waste inventory information could prevent drum opening on the site without prior review.

6.2.2 Explosive Product Survey

If the site is a waste disposal or storage operation, a survey of commercial producers or users of explosives within the area served by the facility must be conducted. The determination of the area covered in this survey is a judgment that should be based on locations of known waste generators that used the facility and geographic location of the site. Agencies that could assist in identifying explosive producers or users are local and state police units, state transportation departments, the U.S. Department of Transportation (DOT), and EPA state hazardous-waste

permit offices. Standard Industrial Classification (SIC) codes can be used to locate producers of explosives from lists of manufacturers available from state commerce agencies, local chambers of commerce, planning agencies, etc.

6.2.3 Site Inspection

A site visit is required prior to planning a drum opening operation. This visit may be in addition to the Reconnaissance Survey. Information on the following should be gathered during the inspection:

1) Site boundaries - fences, roads, natural boundaries, etc.
2) Access points - travel routes on the site.
3) Adjacent land uses - residential, agricultural, public use areas, commercial establishments, schools, natural areas, etc.
4) Power lines, railroads, and public roads close to the site.
5) Container storage areas - provide observational details; describe if drums are jumbled, stacked, piled, arranged in rows, etc. General condition of drums indicates if containers can be grouped according to visual features, contents or any other classification method.
6) Buildings and other site structures, as well as any other disposal areas such as lagoons, landfills, surface piles, etc.
7) Location of water sources.
8) Location of potential staging areas.

In general, the preliminary assessment and site inspection should have been completed prior to involvement in opening and sampling drums. Field characterization should help to establish ambient conditions and identify potential hot spots. This information is to be plotted on the site sketch. Observations from maps and aerial photographs can also be used in compiling the site sketch.

During the site inspection phase, local officials should be contacted to arrange for fire protection and police support during the operation. Interviews should also be conducted with site workers, local officials, and any other people familiar with the site's history.

The central purpose of the background review is to evaluate the risk presented to personnel engaged in drum-opening operations. An assessment of drum contents is most important because it identifies specific risks. However, other site features also affect the hazard potential. Leaking and corroded drums, crowded and poorly organized conditions, and drums of unknown and apparently diverse origins are conditions that require careful planning.

There are no accurate quantitative methods available to evaluate the total danger. Assessment of the danger is subjective and should be done by personnel experienced in field operations at hazardous sites. Good professional judgment is required, and project management must feel that adequate information is available to support a decision to conduct the drum-opening operations.

Any positive indication of shock-sensitive materials that might react or explode requires special consideration. Sites that are suspected or known to contain such materials are to be referred to

the Project Manager for planning for drum opening. In addition, sites that are judged to be unduly hazardous for any other reason should be referred to the Project Manager.

6.3 Container Selection Considerations

The containers selected for opening and sampling will depend on the purpose of the operation and on considerations of safety (i.e., that is a container that may detonate is to be avoided). Even though many drums are found at uncontrolled disposal sites where the content are unknown, it is worthwhile to consider drum markings and types, as well as drum groupings.

When considering drum selection during sampling, the first choice of drums would be those marked with known hazardous materials (trade name, chemical name, empirical formula) or hazardous labeling. Next would be those isolated by themselves or material contained in an exotic metal container (e.g., aluminum, nickel, monel, stainless steel). Then consideration should be given to the unmarked drum piles or stacks. These should be sampled randomly among the various distinguishable drum lots.

When sampling for site characterization purposes, a concerted effort should be made to distinguish drum lots and to get a drum count among the lots. A drum that appears to be characteristic and in the center of all the major drum lots should be sampled first, followed by drums in as many of the smaller lots as practical. Also, if practical, duplicate samples should be taken on major drum lots at either end of a lot to see if the wastes appear to be characteristic all the way through.

On most abandoned waste sites, there is some organization or pattern to the way the material was placed on the site. The pattern is occasionally as detailed as finding the flammable solvents in one area, acids in another, cyanide in another, recoverable metals in a fourth, and so on. Some disposal facilities stencil control numbers on drums to indicate specific lots. Often, if the site was poorly run, the only indication that a group of drums is related will be their color, size, or type.

Typically, waste is shipped to sites in 55-gallon drums on trucks. About 60 to 80 drums are delivered from a given load, depending on the weight of the load. During the initial site inspection, one should look for distinguishing features in an attempt to define the different lots of drums on the site. Often the trade name, chemical name, or empirical formula will be written on the drum. Another distinguishing feature would be drums of exotic metal such as aluminum, nickel, monel, stainless steel, etc. A manufacturing facility will use a specified DOT coded drum, a strange drum size, or a drum with an unusual configuration or adaptation for a particular process line (i.e., center of drum head fill bung, double-sided fill/vent bungs, etc.).

At almost every site that has been receiving waste, there is an isolated group of containers. Approach these with care and try to determine why they were segregated.

In any lot of drums sometimes encountered is an unusual or out-of-place container. This oddball container will not fit the pattern, color, size, etc., of those around it (e.g., it may be the only distended drum among undistended drums or a lined drum among unlined drums).

An attempt should be made to avoid drums that are structurally damaged or if their movement or sampling would endanger a team member. Samples of drums in stacks or piles should not be taken if at all possible.

Before sampling any drums, an external radioactivity scan must be conducted with the results recorded in the Field Logbook. On a site where many different types of containers are present, based on what they can be expected to contain and in increasing order of hazard, the containers should be sampled in the following order:

1) Paper, plastic, cloth, and burlap bags.
2) Glass carboys and jugs (except chemical reagent or laboratory-packed bottles).
3) Fiberboard drums.
4) Plastic and polyethylene carboys and containers.
5) Plastic-lined steel drums.
6) Steel drums.
7) Exotic metal drums.
8) Odd containers (distended, isolated, etc.).

Attachment 2 contains information of the types, sizes, DOT designation, openings, and recommended opening techniques for the various kinds of containers. The DOT designation, which is usually found on the bottom of a drum, can be useful in determining the material of the drum.

6.1 Drum Inspection

Appropriate procedures for handling drums depend on the contents. Thus, prior to any handling, drums should be visually inspected to gain as much information as possible about their contents. The drums should be inspected for the following:

1. Drum condition, corrosion, rust, punctures, bungs, and leaking contents.
2. Symbols, words, or other markings on the indicating hazards (i.e., explosive, radioactive, toxic, flammable), or further identifying the drums.
3. Signs that the drum is under pressure.
4. Shock sensitivity.

Monitoring should be conducted around the drums using instruments such as radiation meters, organic vapor analyzers (OVA) and combustible gas indicators (CGI). Survey results can be used to classify the drums into categories, for instance:

- Radioactive
- Leaking/deteriorating
- Bulging
- Lab packs
- Explosive/shock sensitive
- Empty

All personnel should assume that unmarked drums contain hazardous materials until their contents have been categorized. Once a drum has been visually inspected and any immediate

hazard has been eliminated by overpacking or transferring the drum's contents, the drum is affixed with a numbered tag and transferred to a staging area. Color-coded tags, labels or bands should be used to identify the drum's category based on visual inspection. A description of each drum, its condition, any unusual markings, the location where it was buried or stored, and field monitoring information are recorded on a Drum Sampling Data Sheet (Attachment 3). This data sheet becomes the principal record keeping tool for tracking the drum on-site.

6.2 Container Handling and Staging

Personnel involved in handling and transporting containerized waste shall work in teams containing no fewer than two people. Visual contact shall be maintained between members of the working team at all times. All team members shall be able to communicate between themselves and with the Site Health and Safety Officer by intrinsically safe two-way radio at all times on the work site.

The handling, movement, and transport of drums and other containers should be by use of mechanical equipment only; no drums should be handled manually. Remote drum handling equipment may consist of a grappler equipped backhoe or front-end loader. Drum transportation should be with front-end loaders or forklifts with modified carrying platforms. Portions of equipment that contact drums or canisters should be constructed of non-ferrous metals or contact portions should be coated or lined to preclude spark generation. Handling and transport equipment must be equipped with full frontal and side splash and explosion shields. Class ABC fire extinguishers shall be fitted to the body of each piece of equipment.

When possible, drums or other containers to be sampled should be opened and sampled in place to minimize handling. However, when drums are stacked or are close together, they may have to be moved to prevent sympathetic detonation of, or chemical reaction with, other drums around the one being opened. The main criterion is distance to other drums. A reasonable distance should be maintained to keep the drum to be opened segregated from the others.

Drums or containers exhibiting the following characteristics require special treatment in handling and sampling:

- leaking or deteriorated drums
- bulging drums
- drums containing explosive or shock-sensitive waste
- drums containing radioactive waste
- laboratory packs
- gas cylinders.

When drums are moved, they should be taken to a staging and sampling area that is lined and diked or bermed to control any major spillage. Again, this area should be far enough away from other drums on the site to prevent a chain reaction. Only one container at a time should be placed in the staging area and opened. One crew can be moving and setting up the remote-opening equipment on the next container while another crew is sampling, labeling, and resealing the first container.

During staging, the drums should be physically separated into the following categories: those containing liquids, those containing solids, those containing lab packs, and those which are empty.

This is done because the strategy for sampling and handling drums/containers in each of these categories or deformation) and their contents are known to be will be different. This may be achieved by visual inspection of the drum and its labels, codes, etc. Solids and sludges are typically disposed of in open top drums. Closed head drums with a bung opening generally contain liquid.

Where there is good reason to suspect that drums contain radioactive, explosive, or shock-sensitive materials, these drums should be staged in a separate isolated area. Placement of explosives and shock- sensitive materials in diked and fenced areas will minimize the hazard and the adverse effects of any premature detonation of explosives.

Where space allows, the drum opening area should be physically separated from the drum removal and drum staging operations. Drums are moved from the staging area to the drum opening area one at a time using forklift trucks equipped with drum grabbers or a barrel grappler. In a large-scale drum handling operation, drums may be conveyed to the drum opening area using a roller conveyor. Drums may be restaged as necessary after opening and sampling.

Containers that are inside warehouses, basements, or other buildings must be moved outside before they can be opened. Adequate ventilation is critical for container-opening operations.

Empty drums containing less than one inch of solid residual waste, and those resulting from on-site bulking and repack operations, shall be loaded by grappler into transport equipment and placed within the empty drum staging area. Residuals, where possible, shall be transferred to repack containers prior to movement.

6.3 Remote Opening

Because of the possibility of encountering a drum containing a shock-sensitive material, any drum to be moved and/or sampled should be remotely shaken. One way of doing this is to carefully tie a rope around the drum and shake it from behind a barrier at a safe distance.

The required method of opening drums is by remote means, except as noted in Attachment 2. Three types of remote-opening equipment are available: the bung spinner, the remote-controlled drill, and the drum pierce. The bung spinner consists of:

1) Air impact wrench with non-sparking adapter
2) Drum-mounting bracket
3) Two-stage regulator
4) Compressed air cylinder with 100 feet of air hose and control valve.

The impact wrench is mounted over the bung on top of the drum by means of the steel-mounting bracket. The air tank, regulator, and control valve can be placed up to 100 feet away from the drum in a well protected location.

A remote-controlled, air-operated, self-feeding, and self-retracting drill can also be used. This tool consists of:

1) Self-feeding and self-retracting drill
2) Drum-mounting bracket
3) 100 feet of air and control hoses
4) Two-stage high-pressure regulator
5) Compressed air cylinder
6) Filter/regulator/lubricator unit.

As with the bung spinner, the air tank, regulator, and control valves can be placed up to 100 feet away from the drum in a well-protected location. There are two controls on this piece of equipment, a start valve and an emergency retract valve. The drum non-sparking pierce consists of:

1) Hydraulic ram with hand pump
2) 100 feet of hydraulic hose
3) Drum-mounting bracket (top or side)
4) Piercing nail.

This unit uses the same bracket as the drum drill. The hydraulic ram slowly forces a non-sparking pierce through the drum surface as the hand pump is operated. When the 1/2-inch diameter hole is complete, opening a relief valve on the pump allows the spring to retract the piercer from the hole.

When any of these pieces of equipment is used, the control lines are to be extended to their maximum, and drum-opening personnel are to operate the controls from behind sandbags, concrete or brick structure, or other solid barriers. Remember, the opening surfaces of the drill or bung spinner should be decontaminated after each use.

The following guidelines are offered for other types of containers:

1) **Ring-closed, open-top drums** - Loosen the ring and then remove it remotely by means of a rope. If it is necessary to cut the ring, do so near the bolt or clamp/lever so that there will be a place to attach the rope.
2) **Glass carboys or jugs with lapped/ground-glass top or plastic cap** - Slowly release any retaining wire and vent any pressure. Remove the stopper or cap by hand only.
3) **Fiberpacks or corrugated cardboard containers** - Release the locking ring and remove the ring and lid by hand.
4) **Plastic or polyethylene carboys and plastic-lined drums, when necessary** - Use a nonsparking aluminum, brass, or beryllium bung wrench of the proper size. Do not use a bung wrench on any distended drums of this type; remote methods will be applied.
5) **Plastic Kraft paper, burlap, or cloth bags** - Use a trowel or sampling trier. The bags should be resealed or placed in an overpack.

6.4 Problem Containers

Special handling techniques are required for containers which may expose personnel to particularly hazardous conditions. These techniques are described in general below, although site-specific conditions may require the development of specialized methods.

6.4.1 Leaking or Deteriorated Drums

1) The contents of drums that exhibit leakage or apparent deterioration such that movement will cause rupture (determined by the Health and Safety Officer) must immediately be transferred to a repack drum. Equipment, including transfer pumps used in the repack operation, must be of explosion proof construction.
2) Leaking drums containing sludge or semi-solids, drums that are structurally sound but which are open and contain liquid or solid waste, and drums which are deteriorated but can be moved without rupture must be immediately placed in overpack containers.

6.4.2 Bulging Drums

1) Drums which potentially may be under internal pressure, as evidenced by bulging, must be sampled in place. Extreme care shall be exercised when working with and adjacent to potentially pressurized drums.
2) Should movement of a pressurized drum be unavoidable, handle only by a grappler unit constructed for explosive containment. The bulging drum should be moved only as far as necessary to allow seating on firm ground or it should be carefully overpacked.
3) Openings into pressurized drums shall be plugged and the bung holes fitted with pressure venting caps set at 5 psi release.

6.4.3 Drums Containing Explosive or Shock Sensitive Waste

1) If drums containing wastes that have been identified by sampling, or are suspected by visual examination to be explosive in nature are found, the Project Manager and the Health and Safety Officer must be notified immediately, before the drums are handled in any way.
2) If the Project Manager and the Health and Safety Officer approve handling of these drums, they shall be handled with extreme caution. Initial handling shall be by a grappler unit constructed for explosive containment. Drums shall be palletized prior to transport to a high hazard interim storage and disposal area.
3) If at any time during remedial activities, an explosive, pursuant to provisions of Title 18, U.S. Code, Chapter 40 (Importation, Manufacturer, Distribution, and Storage of Explosive Materials, 1975 Explosives List) is identified, it should be secured and the appropriate state and federal agencies notified.
4) Identification of an explosive substance during the course of a remedial action is usually based on the experience of the on-site personnel. Potentially explosive materials usually may be identified by their physical characteristics (i.e., texture, color, density, etc.), as well as the way they are packaged or labeled. Most explosives are solids. In some cases they are packaged in water-tight containers to exclude water, while in other cases they are packaged wet to preclude explosion.
5) Prior to handling or transporting drums containing explosive wastes, personnel working in the area shall be removed to a safe distance as determined by the Health and Safety Officer. Continuous contact with the communication base shall be maintained until handling or transporting operations are complete. An audible siren signal system,

similar to that employed in conventional blasting operations, shall be used to signify the commencement and completion of explosive waste handling or transporting activities.

6.4.4 Drums Containing Radioactive Waste

1) Drums containing radioactive wastes shall not be handled until radiation levels have been determined by an initial field survey which is recorded in a Field Logbook. The survey shall include background levels, direct gamma readings and laboratory analysis of drum surface wipe samples.
2) Depending on the level of radiation encountered, handling and transport may require special shielding devices to protect personnel. Following handling and transport, equipment used shall be surveyed by the Health and Safety Officer and decontaminated to background levels prior to recommencing work. Surveys shall also be made of the ground surface in the vicinity of original drum storage to identify potential soil contamination by spilled or leaked radioactive waste. Prior to recommencing work in the area, radioactive soil areas shall be isolated to prevent tracking of radioactive contaminants about the site, and workers who entered the area should have their gloves

6.4.5 Packaged Laboratory Wastes (Laboratory Packs)

1) If drums known or suspected of containing discarded laboratory chemicals, reagents or other potentially dangerous materials in small volume, or individual containers are found, the Project Manager is to be notified immediately, before the drums or containers are moved or opened.
2) If the Project Manager and the Health and Safety Officer approve the handling of these containers, they shall be handled with extreme caution. Until otherwise categorized, they shall be considered explosive or shock-sensitive wastes. Initial handling shall be by a grappler unit constructed for explosive containment. Drums shall be palletized and over-packed if required prior to transport to the Laboratory Pack staging area for sorting, identification, repacking and/or stabilization.
3) Prior to handling or transporting Laboratory Packs from the existing drum area, personnel working in the immediate area shall be removed to a safe distance. Continuous contact with the communication base shall be maintained until handling or transporting operations are complete. An audible siren signal system, similar to that employed in conventional blasting operations will be used to signify the commencement and cessation of Laboratory Pack handling or transporting activities.

6.4.6 Air Reactive Wastes

1) If the presence of air reactive substance is verified or even suspected, the material should be immediately segregated and transported to a separate high hazard interim storage and disposal area.
2) Air reactive wastes may be discovered during opening or sampling operations. Air reactive substances normally require special packaging. They may be stored under water or some other liquid to minimize air contact. They may also be found in sealed ampoules, corrugated drums, stainless steel canisters, or specially lined drums.

6.4.7 Gas Cylinders

Gas cylinders, when encountered, should be stored and disposed of on a special case basis depending on the integrity of the cylinders and type of substance they are expected to contain.

6.5 Container Sampling

6.5.1 Equipment

1) Personal protection equipment.
2) 500 ml, wide-mouth amber glass bottle with Teflon® cap liner.
3) Uniquely numbered sample identification labels and tags, filled out and affixed to sample containers before sampling commences.
4) Four-foot long by 3/4 inch inside diameter glass sampling thief.
5) Remotely operated opening device (see Section 6.5).
6) One gallon covered cans half-filled with absorbent (for off-site shipment only).

6.5.2 Glass Thief Sampling Procedures

All drums and mechanical equipment should be grounded prior to the commencement of sampling. If the bung or container lid can be removed, sample contained liquids using a glass thief, which shall then be broken and discarded within the barrel. A barrel that has a badly rusted bung, or that cannot be sampled as above, shall be safely entered with a hydraulic penetrating device operated remotely (see Section 6.5). All openings shall be plugged except during sampling operation. The steps to be followed in sampling are as follows:

1) Record any markings, special drum conditions, and type of opening in the Field Logbook, on the sample log sheet, and, later, on the Chain-of-Custody form. Locate the general area on a sketch of the site.
2) Stencil an identifying number on the drums and record in the logbook. Consult the project-specific work plan for identifications.
3) Make certain that the drum/container is set on a firm base, preferably in a fully upright position.
4) Open the drum/container as described in Section 6.5 and Attachment 2.
5) Insert glass tubing almost to the bottom of the drum or until a solid layer is encountered. About one foot of tubing should extend above the drum.
6) Allow the waste in the drum to reach its natural level in the tube. Cap the top of the sampling tube with a tapered stopper, ensuring liquid does not come into contact with stopper.
7) Carefully remove the capped tube from the drum and insert the uncapped end in the sample container. Release the stopper and allow the glass thief to drain completely into the sample container.
8) Deliver 100 to 250 ml of the sample to a clean, wide-mouth, 500-ml glass sample jar. If the sample is not free flowing and is taken through a bung opening, repeated sampling may be necessary.
9) Place the used sampling tube, along with paper towels or waste rags used to wipe up any spills, into an empty metal barrel for subsequent disposal. If glass tubing has been used, it may be broken and left inside the drum being sampled.
10) Clamp the sample container tightly and place pre-labeled and tagged sample container in a carrier.
11) Replace the bung or lids or place plastic over the drum/container.
12) Measure the sample for radioactivity and record results in a Field Logbook. If the meter readings exceed 10 mRhr, notify the Field Team Leader immediately.
13) Fill out Chain-of-Custody Record and carefully pack samples. The finished package will be padlocked or custody-sealed for shipment to the laboratory. The preferred procedure

includes the use of a custody-seal across filament tape that is wrapped around the package at least twice. The custody-seal (paper, plastic, or metal) is folded over and stuck to itself so that the only access to the samples is by cutting the filament tape or breaking the seal to unwrap the tape. The seal is signed before the package is shipped.

14) Complete the appropriate traffic report. Drum samples are always considered to be high-hazard samples.

6.5.3 Coring Device Sampling Procedure

1) Record any markings, special drum conditions, and type of opening in the Field Logbook, on the sample log sheet, and, later, on the Chain-of-Custody form. Locate the general area on a sketch of the site.
2) Stencil an identifying number on the drums and record in the logbook. Consult the project-specific work plan for identifications.
3) Make certain that the drum/container is set on a firm base, preferably in a fully upright position.
4) Open the drum/container as described in Section 6.5 and Attachment 3.
5) Insert the coring sample device into the drum solids.
6) Rotate the sampling device to cut a core of the material.
7) Slowly withdraw the sampling device so that as much of the sample material as possible is retained.
8) Transfer the sample to the appropriate sample container, and label it. A stainless spoon or scoop may be used as necessary.
9) Clamp the sample container tightly and place pre-labeled and tagged sample container in a carrier.
10) Replace the bung or lids or place plastic over the drum/container.
11) Fill out Chain-of-Custody Record and carefully pack samples. The finished package will be padlocked or custody-sealed for shipment to the laboratory. The preferred procedure includes the use of a custody-seal across filament tape that is wrapped around the package at least twice. The custody-seal (paper, plastic, or metal) is folded over and stuck to itself so that the only access to the samples is by cutting the filament tape or breaking the seal to unwrap the tape. The seal is signed before the package is shipped.

6.5.4 Sample Preservation and Packing Procedures for Drummed Waste Samples

1) No preservatives shall be used.
2) Place sample in a Ziplock plastic bag.
3) Place each bagged container in a 1-gallon covered can containing absorbent packing material. Place lid on can.
4) Mark the sample identification number on the outside of the can.
5) Arrange for the appropriate transportation mode consistent with the type of hazardous waste involved.

6.6 Resealing and Restage Containers

1) All containers opened for sampling need to be resealed to prevent the escape of vapors and possible reactions from rainwater, air and so on. The resealing methods will depend on the opening methods used and include the following:
2) Replacing the bung, screw cap, etc.
3) Replacing the lid and retaining ring.

4) Placing the drum in an overpack (larger drum) when it cannot be resealed by any other method.
5) If a hole is drilled, use a special rubber plastic plug. A drum bonnet should be used to ensure that rainwater does not seep around the plug.

It is important to note that these resealing methods are for the purpose of preventing leakage from the container while it is in storage on the site. If the container is to be moved off the site, DOT regulations regarding transportation of drums must be complied with. These will generally require more rigorous sealing procedures.

Once the drum is sampled and resealed, it should be left where it cannot react with other containers on the site. For a small number of drums, the storage areas may be the staging and opening area. In any event, the sampled drums should be placed in an area away from other groups of containers on the site. The reason is that slowly progressing chemical reactions can start when a container is opened and the contents exposed to air or the disturbance caused by handling the drum. Such a reaction could take hours or even days to occur.

6.7 Public Evacuation/Alert Consideration

6.7.1 General

The potential need for evacuation of the site and surrounding area must be considered. Several site-specific factors influence the need for, and the extent of, the evacuation or alerting of the nearby off-site public. These factors include the following:

1) Proximity of residences, shopping or other commercial or business areas, factories, highways, railroads, and airfields or other transportation routes that may have to be evacuated. This information will be available from the background review and preliminary site inspections.
2) Proximity of other facilities that could be involved in, cause or propagate a fire, explosion, or toxic release on the site. This information will also be known from the background review and site inspection.
3) Presence of explosive, flammable, or volatile substances on the site. Some general indications of the types of hazards present may be provided by the background review and site inspection. The probability of encountering explosives (i.e., directly detonatable or shock-sensitive materials as opposed to explosive vapor-oxygen mixtures) will have been reduced by the screening procedures applied during earlier site evaluation. Preliminary assessment and site inspection may provide indications, or definite knowledge, that specific compounds presenting known flammability or toxicity hazards are in the containers. Of these known hazards, those having the greatest potential for atmospheric spread off the site should be used in estimating evacuation hazard distances as described below. For example, if several volatile toxic liquids, or toxic vapors, are present, those having the greatest toxic potential in air, as measured by a Threshold Limit Value (TLV) or classified as Immediately Dangerous to Life and Health (IDLH), should determine the hazard distance, since these have the potential for the greatest health impacts.

   Atmospheric drift of a toxic or flammable vapor cloud or plume can often extend to great distances from the site, and hence potentially threaten more people than even an

explosive hazard. Similarly, thermal-radiation hazards generated by even a large fire on the site generally reach to distances which are small compared to possible atmospheric drift distances of a vapor cloud.

4) Potential for an accident on the site which could result in an atmospheric release of flammable or toxic liquid or vapor. This possibility should be remote if only one drum is opened at a time and if that drum is segregated from the other drums.

The most important parameter that needs to be established for any accident is the rate of liberation of flammable or toxic vapor; unfortunately, this is often the most uncertain quantity.

5) Prevailing wind speed and direction and atmospheric stability strongly affect the pattern of atmospheric spread of a gas cloud. If these can be quantitatively estimated at the time an actual accidental release occurs, this information should be used in calculating and estimated evacuation corridor as detailed below.

However, because wind direction is subject to rapid and unpredictable variations, and because atmospheric drift of a concentrated cloud or plume is greatest under stable atmospheric conditions and low winds speeds, it is usually preferable to take a conservative approach. Thus, one should base a public hazard evacuation distance, in any direction from the site, on an assumed worst-case atmospheric condition, that is, a stable atmosphere and nominal low wind speed, 5 mph.

6.7.2 Plausible Accident Scenario

A plausible but hypothetical scenario for an accident that may be expected to occur during closed container opening operations would involve a release, from only the one 55-gallon drum being opened, of a volatile toxic liquid that rapidly vaporizes and forms and non-burning but continuous source of a toxic vapor plume. The rate of vapor generation and release can be calculated from the assumption that the upright drum is completely open at the top and knowledge of the vapor pressure and some other readily available chemical properties of the chemical involved. For simplicity, the fact that a complex mixture of chemicals may actually be involved is neglected and the most toxic liquid or vapor is treated as if it were a pure component.

6.7.3 Estimating Hazard Evacuation Radius

Once the rate of atmospheric release of vapor is estimated for the accident scenario, outside assistance from any of several sources may be sought to estimate an atmospheric dispersion distance appropriate for the degree of flammability or toxicity hazard of the chemical involved. This estimate would then be used as a recommendation of an evacuation radius to be made to the responsible official in charge at the site, who will actually determine the necessity and extent of public evacuation. Outside assistance in estimating the hazard radius in an emergency situation may be obtained from EPA's Emergency Response Team (ERT), the U.S. Coast Guard's Hazard Assessment Computer System (HACS), or from other hazard analysts.

Two different situations may require the evacuation of the off-site public:

1) The emergency resulting from an actual occurrence of an accident involving atmospheric release during drum-opening operations.

2) Precautionary planning before the start of drum-opening operations, in anticipation of an accident.

The above hypothetical scenario involving a single drum may be used in planning precautionary evacuations before the start of a dangerous drum-opening operation. On the other hand, in an actual accident, the release rate may be estimated if the number of drums releasing and the size of the opening in each such drum can be estimated by observation.

The decision to evacuate or alert the public off the site as a precautionary measure depends on the degree of hazard presented by the materials known to be present at the site. For the scenario described above, a table of numerical hazard distances for several of the commonly encountered chemicals and those expected to be found at the site should be prepared before drum-opening commences. These distances may then be used as numerical decision criteria for precautionary evacuation by comparing them to the known distances of populated areas from the site.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

NUS Corporation, 1983. *Operating Guidelines Manual.*

Cassic, J.A., et al., 1985. *Guidance Document for Cleanup of Surface Tank and Drum Sites.* Prepared for Office of Emergency and Remedial Response, USEPA, Washington, DC under Contract No. 68-01-6930.

IT Corporation, December 1988. *Hazardous Waste Operations and Emergency Response*, IT Corporation, Knoxville, Tennessee.

Martin, F.M., Lippitt, J.M., Prothero, T.G., 1987. *Hazardous Waste Handbook for Health and Safety*, Butterworth Publishers, p. 167-177.

8.0 DEFINITIONS

Air Reactive Wastes - Some chemicals, such as white phosphorus or barium oxide, react with oxygen in the air, while others, such as sodium, cesium or various metal hydrides, react with the moisture or water vapor in the air. Many of these compounds are explosive when they come in contact with air or water.

Container - Defined as any drum, bottle, can, bag, etc., with a capacity of 120 gallons (450 liters) or less.

Glass Thief - A glass tube 4 feet long and 3/4 inches in diameter, used for taking samples from drums. The tube is usually broken and disposed of in the drum following sampling.

Polyethylene or PVC-lined Drums - Often contains strong acids or bases. If the lining is punctured, the substance usually corrodes the steel, resulting in a significant leak or spill.

Exotic Metal Drums (i.e., aluminum, nickel, stainless steel, or other unusual metals) - Very expensive drums that usually contain an extremely dangerous material.

Single-Walled Drums Used as a Pressure Vessel - These drums have fittings for both product filling and placement of an inert gas, such as nitrogen. Such drums may contain reactive, flammable, or explosive substances.

Laboratory Packs - Such drums are commonly used for disposal of expired chemicals and process samples from laboratories, hospitals and similar institutions. Bottles in the laboratory pack may contain incompatible materials and may not be packed in absorbent material. They may contain radioisotopes, shock sensitive, highly volatile, highly corrosive, or very toxic exotic chemicals. Laboratory packs are the primary ignition source for fires at most hazardous waste sites.

9.0 ATTACHMENTS

1) Reference Information for Procuring Qualified Subcontractors for Drum Handling and Removal.

2) Techniques for Opening Containers

3) Drum Sampling Data Sheet

Attachment 1

Reference Information for Procuring Qualified Subcontractors for Drum Handling and Removal

1.0 PURPOSE

The purpose of this attachment is to provide reference information for procuring a qualified subcontractor for drum handling and removal activities at hazardous waste sites.

2.0 SCOPE

It is assumed that the removal and/or special on-site handling of drums at a hazardous waste site will require the services of a subcontractor specialist and that the lead firms within ERT will not undertake this activity themselves. This attachment is limited to addressing the procurement of a qualified subcontractor for drum handling and removal activities.

3.0 REQUIREMENTS

Drum handling and removal is normally handled by specialty subcontractors. Specific contractual requirements are necessary to address this dangerous and sensitive activity.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for identifying the need for procuring drum handling and removal services and for developing the bid package, technically reviewing bids and preparing the purchase requisition.

4.2 Field Team Leader

The Field Team Leader is responsible for monitoring the progress of the drum handling/removal subcontractor while the subcontractor is on-site, establishing that the subcontractor conforms to the requirements of the work scope.

5.0 PROCEDURES

5.1 Information to be Transmitted to the Subcontractor

In preparing the bid package, the Project Manager shall provide all available information as related to the drum handling activity in question. The request shall make it a contractual requirement that the same information be transmitted by the main subcontractor to any lower-tier subcontractors who may be needed in order to complete the task. The information shall include, but is not limited to, the following:

- General information and project/site background
- Scope of work and proposed date(s) of activity
- Waste characterization including:
  1) Drum contents
  2) Numbers of drums
  3) Physical condition of drums
  4) Physical description of drums.
- Known chemical and physical hazards associated with the site and drums
- Health, Safety and Training Requirements including, but not limited to the following:

1) Health and Safety Plan (HASP) for the AEC Program which defines:
   a. Medical surveillance requirements for all subcontractor personnel. Prior to subcontractor
   b. Personnel performing on-site work, each person must successfully complete, at subcontractor expense, the medical monitoring defined in the HASP and must provide the specified Physical Statement signifying medical approval to perform site work.
   c. Fundamental Health and Safety Training (1 day) which is to be completed by the subcontractor's on-site employees prior to site work and which will generally be provided by the Health and Safety Officer assigned to the site.
2) Personal Protective Equipment requirements, which should be defined in the project specific HASP prepared by the Health and Safety Officer.
3) Contract language requirements for the subcontractor to comply with all requirements of the Program HASP, the project-specific work plan, and all applicable Federal, State and Local Health and Safety regulations. Contract language should also specify that the Health and Safety Officer may stop the subcontractor's work on his failure to comply with any of these requirements and that subsequent damages may be assessed.

5.2 Information Required to Evaluate the Subcontractor

- The subcontractor and any lower-tier subcontractor to be used should be required to provide the following information as part of their bid:
- Complete case history regarding drum handling activities on hazardous waste sites and references regarding job performance
- Experience of specific subcontractor personnel who are to perform the drum handling activity in question
- Detailed description of equipment to be used for performing the scope of work. The bid package should request sufficient information from the bidders to permit the following considerations to be addressed:
- Experience and Reference
  1) How long has the subcontractor been in business?
  2) How many similar drum handling jobs has the subcontractor performed?
  3) Are references favorable?
  4) What is the experience of personnel specifically assigned to this project?
  5) Are the personnel in a current medical monitoring program?
  6) Is the subcontractor familiar with Health and Safety training and Operational Procedures?
- Training
  1) Does the subcontractor have an employee training program?
  2) If so, how often is refresher training given?
  3) Are personnel certified or licensed by reputable agencies, associations?
  4) What does the training program encompass? Equipment operation? Health and Safety? Proper working procedures?
- Procedures
  1) Are the subcontractor's drum handling procedures consistent with EPA protocol, OSHA procedures, and other applicable standards?
  2) Do these procedures account for:

a) environmental stress?
b) inclement weather?
c) upgrading in the level of protection?

3) How does the subcontractor plan to carry out these procedures?
a) how many operators?
b) how many helpers?
c) what are the specific responsibilities for assigned personnel?

- Equipment
1) Is the subcontractor's equipment:
a) in conformance to OSHA standards?
b) regularly inspected?
c) regularly serviced?
d) easily serviceable?
e) modern?
2) Does the subcontractor employ:
a) drum grapplers attached to a hydraulic excavator?
b) front-end loader, which can be loaded manually, equipped with a bucket sling, or used with a right terrain forklift?
3) Which of the aforementioned methods is better for the job?
4) Has the subcontractor proposed another acceptable method which is more feasible?
5) Has the subcontractor attempted to employ remote handling equipment that will lessen the potential for worker contact? Or is all work manual, presenting improper lifting and potential back injury concerns?
6) Does loading equipment contain:
a) an air conditioned cab for operation safety and comfort?
b) an overhead canopy and safety splash shield for operation safety?
c) emergency escape packs if this is deemed necessary?
d) provision for use of supplied air respirators?

- Costs
Are costs:
1) Clearly outlined for the scope of work?
2) Responsible and competitive for the procedures and equipment being used?

These criteria should be cited as part of the detailed bid package for technical evaluation and selection of a drum handling subcontractor.

Attachment 2

Techniques for Opening Containers

Types	Size	DOT Designation	Type of Opening	Recommended Opening Technique
Steel drum, open head, unlined and lined	5-11OG	DOT 5-DOT6-DOT17-DOT37-DOT42-	Detachable steel lid with a clamp or lever-locking ring, or a ring with forged lugs and secured by a bolt.	Remove bolt. If possible, relieve pressure on clamp or lever-locking ring remotely (i.e., lanyard); Remove ring with layard. Remove lid by hand.
Steel drum, closed head, lined	5-11OG	DOT5-DOT6-DOT17-DOT37-DOT42-	Plastic bung opening not larger than 2.3 in.	Preferred method is to remotely open bung. Manually open otherwise.
Steel drum, closed head, unlined (steel, monel, stainless, nickel, and aluminum)	5-11OG	DOT5-DOT6-DOT17-DOT37-DOT42-	Steel or other metal bung not over 2.3 in.	Remote method.
Burlap bag, double Kraft paper bag, cloth bag, plastic bag	Various	DOT36-DOT44-DOT45-	Various	Open with sharp implement; reseal bag or overpack in fiberpack.
Glass carboys and jugs	6-20G	Usually DOT 1-branded into the wooden outer sheathing; often sheathing is no longer present.	Lapped or ground glass stopper; occasionally a plastic screw cap will be encountered.	Manually.
Laboratory reagent bottles (amber bottles), small reagent cans	Various	None	Screw top or press lid.	Usually encountered in lab packs. Not to be handled or sampled. Replace drum lid carefully. Contact Project Manager for action.
Polyethylene and other plastic drums or barrels	5-11OG	DOT2-	Usually bung opening not over 2.7 in. in diameter.	Manually.
Gas Cylinders	Various	DOT3- DOT4-DOT8-DOT39-	Valve, threaded fitting, quick-connect or puncture- type fittings.	Not to be handled or sampled. Contact Project Manager for action.
Fiberpack or corrugated	5-11OG	DOT12-DOT21-DOT23-	Usually a detachable plastic lid with a clamp or lever- locking ring.	Manually.

Attachment 3

Drum Sampling Data Sheet

Samplers:_____Date:_____

Site Name:_____ Project Number:_____

Container Number/Sample Number: REAC Task Leader:

SITE INFORMATION:

Terrain, drainage description:_____

Weather conditions (from observation):_____

MET station on site: No Yes

CONTAINER INFORMATION:

Container type: Drum Tank Other:_____

Container dimensions: Shape:_____

Approximate size:_____

Label present: No Yes:_____

Other Markings: _____
_____
_____
_____

Spill or leak present: No Yes Dimensions:_____

Container location: (Circle one) N/A See Map Other: _____
_____
_____
_____
_____
_____

Attachment 3 (Cont'd)

Drum Sampling Data Sheet (Cont'd)

SAMPLE INFORMATION:

Description: _____ liquid _____ solid (_____ powder or _____ crystals) _____ sludge

Color: _____ Vapors:_____

Other:_____

Local effects present: (damage - environmental, material)_____

FIELD MONITORING:

PID: _____ Background (clean zone)
_____ Probe used/Model used
_____ Reading from container opening

FID: _____ Background (clean zone)
_____ Reading from container opening

Radiation Meter:
_____ Model used
_____ Background (clean zone)
_____ Reading from container opening

Explosimeter/Oxygen Meter:
_____ Oxygen level from container opening
_____ LEL level from container opening

SAMPLE DESCRIPTION:

_____ Liquid _____ Solid _____ Sludge _____ Color _____ Vapors

WATER REACTIVITY:

Add small amount of sample to water: _____ bubbles _____ color change to _____
_____ vapor formation _____ heat _____ No Change

SPECIFIC GRAVITY TEST (compared to water):
Add small amount of sample to water: _____ sinks _____ floats

If liquid sample sinks, screen for chlorinated compounds. If liquid sample floats and appears to be oily, screen for PCBs (Chlor-N-Oil kit).

Attachment 3 (Cont'd)

Drum Sampling Data Sheet (Cont'd)

CHLOR N OIL TEST KIT INFORMATION:

Test kit used for this sample: Yes No

Results: _____ PCB not present _____ PCB present, less than 50 ppm

_____ PCB present, greater than 50 ppm _____ 100% PCB present

WATER SOLUBILITY TEST:

Add approximately one part sample to five parts water. You may need to stir and heat gently. **[DO NOT HEAT IF WATER REACTIVE!]** Results: _____ total _____ partial _____ no solubility

pH OF AQUEOUS SOLUTION:

Using 0-14 pH paper, check pH of water/sample solution: _____.

SPILL-FYTER CHEMICAL CLASSIFIER STRIPS:

Acid/Base Risk: (Circle one)	Color Change
Strong acid (0)	RED
Moderately acidic (1-3)	ORANGE
Weak acid (5)	YELLOW
Neutral (7)	GREEN
Moderately basic (9-11)	Dark GREEN
Strong Base (13-14)	Dark BLUE
Oxidizer Risk: (Circle one)	
Not Present	WHITE
Present	BLUE, RED, OR ANY DIVERGENCE FROM WHITE
Fluoride Risk: (Circle one)	
Not Present	PINK
Present	YELLOW

Attachment 3 (Cont'd)

Drum Sampling Data Sheet (Cont'd)

Petroleum Product, Organic Solvent Risk: (Circle one)

Not Present	LIGHT BLUE
Present	DARK BLUE

Iodine, Bromine, Chlorine Risk: (Circle one)

Not Present	PEACH
Present	WHITE OR YELLOW

SETAFLASH IGNITABILITY TEST:

140$_o$F	Ignitable: _____	Non-Ignitable _____
160$_o$F	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____

Comments:
_____
_____
_____
_____
_____
_____

HAZCAT KIT TESTS:

Test: _____ Outcome:_____
Comments:_____
_____

Test: _____ Outcome:_____
Comments:_____
_____

Attachment 3 (Cont'd)

Drum Sampling Data Sheet (Cont'd)

Test: _____ Outcome:_____
Comments:_____
_____

Test: _____ Outcome:_____
Comments:_____
_____

Test: _____ Outcome:_____
Comments:_____
_____

HAZCAT PESTICIDES KIT:
Present: _____ Not Present: _____
Comments: _____
_____
_____

Earth Resources Technology, Inc. Standard Operating Procedure **GROUNDWATER LEVEL MEASUREMENT**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-07-2**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
 Signature	1/22/09 Date	 Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to provide general reference information and technical guidance on the measurement of static groundwater.

2.0 SCOPE

This procedure gives overall technical guidance for obtaining groundwater level measurements in monitoring wells frequently conducted in conjunction with groundwater sampling events.

3.0 REQUIREMENTS

Groundwater level measurements can be made in monitoring wells, private or public water wells, peizometers, open boreholes, or test pits (after stabilization). Groundwater measurements will not be made in boreholes with drilling rods or auger flights present, as applicable. If a groundwater elevation measurements must be take while objects are present, the value is assumed to be an estimated groundwater level. If groundwater sampling activities are to occur, groundwater level measurements will be conducted prior to well purging or sampling.

4.0 RESPONSIBILITIES

4.1 Field Team Leader

The Field Team Leader is responsible for obtaining valid groundwater level measurements in accordance with this procedure. All collected data will be reviewed by the Field Team Leader for accuracy and transferred to the Project Manger for inclusion in the project file.

4.2 Field Team

The Field Team is responsible for obtaining valid groundwater level measurements in accordance with this procedure and will be properly trained in the use of necessary equipment. The Field Team is responsible for conducting groundwater level measurements in accordance with the project-specific requirements in the Sampling and Analysis Plan (SAP).

5.0 EQUIPMENT

The equipment used to gauge groundwater level may include the following:

- Electronic Water Level Indicator (graduated to 0.01 feet)
- Interface probe (graduated to 0.01 feet)
- Portable Photoionization Detector (PID)
- Deionized Water and phosphate-free soap for decontamination of the Water Level Indicator and/or Interface Probe
- Field Logbook

6.0 PROCEDURE

- Initial monitoring of the well headspace and breathing zone concentrations using a PID shall be evaluated by field personnel to determine required levels of protection.
- All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the Field Logbook.
- A clearly-established reference point of known altitude, which is normally identified by a mark along the upper edge of the inner well casing or a small notched cut into the well casing, will be used as the dedicated measuring point for gauging groundwater levels. If a survey mark is absent, field personnel will determine to the best of their ability the highest point on the casing and make an indelible mark with a permanent marker or create a distinguishable notch in the well casing for future gauging activities. The absence of the mark and associated corrective action will be reported to the Project Manager and recorded in the Field Logbook.
- Soon after a monitoring or groundwater observation well has been installed and the groundwater level has stabilized, the date and time of the gauging, the initial depth-to-water and total depth of the well will be measured and recorded in the field logbook.
- Cascading water within a borehole can cause false readings. Non-aqueous phase liquid layers may also cause problems in determining the true water level in a well by artificially depressing the true potentiometric surface of the groundwater.
- All groundwater level measurements collected from a site should be obtained in the shortest possible time in order to minimize variations in groundwater elevations caused by external forces (i.e. tidal influences, precipitation events).

6.1 Water Level Measuring Techniques

Water level measurements will follow this sequence:

1. Check the operation of recording equipment above ground. This can be accomplished by filling a container with water and slowly lowering the probe into the water. If the probe recognizes the water interface, the measurements from the monitoring well can be assumed to be accurate.

2. All groundwater level measurement devices will be decontaminated in accordance with ERT SOP-03-1 prior to and after use at each location to prevent cross-contamination between monitoring wells.

3. Slowly lower the water level indicator into the well until static water is encountered. Once encountered raise the indicator out of the water and slowly reestablish contact with the groundwater surface. Repeat this process as necessary in order to accurately record the groundwater depth to within 0.01 feet.

Record all information specified below in a Field Logbook:

- If non-aqueous phase liquids (both LNAPL and DNAPL) are suspected to be present an interface probe will be used to determine the presence and thickness of any measurable layers. Measurements will be recorded to the nearest 0.01 foot.
- If NAPL is not anticipated, an electronic water level meter can be used to measure the groundwater level and will be recorded to the nearest 0.01 foot.

- Record date and time, well number, depth to water, depth to non-aqueous phase liquids (if present), total depth, well diameter, and volume of standing water column in the well in the field logbook. Recording this data in a table format facilitates easier evaluation and transference of data from the Field Logbook.

6.2 Water Level Measuring Devices

6.2.1 Electronic Water Level Indicators

These devices consist of a spool of flat plastic tape with encased electrical wires connected to a contact probe at the end. When the probe comes in contact with the water, an electrical circuit is closed and a light, and/or buzzer attached to the spool will signal the contract. In conditions where there is oil on the water, ground water with high specific conductance, water cascading into the well, or turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well. The electric tape is graduated on the tape surface to 0.01 feet. Once the device signals the depth to water the tape should be move to the side of the casing where the permanent measurement point is marked on the well casing. The measurement is then read and the recorded in the Field Logbook.

6.2.2 Interface Probe

Interface probes work similar to electronic water level meters except that there are an additional set of electrical contacts on the tip of the contact probe. The conductivity of non-aqueous-phase liquids, such as petroleum products, differs from ground water. The interface probe is sensitive to the conductivity difference and when the probe contacts non-aqueous phase liquids the probe emits a unique audible alert indicating the presence of such a liquid. The tape is graduated to 0.01 feet.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operating Procedure:

Fetter, C.W., 1988 *Applied Hydrogeology* Prentice Hall, Inc., Upper Saddle River, New Jersey.

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Environmental Protection Agency (USEPA), 2007. *Field Branches Quality System and Technical Procedures*. Region 4 Science and Ecosystem Support Division. SESDPROC-105-R1.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology,* McGraw-Hill, New York

8.0 DEFINITIONS

Artesian Conditions - A common condition in a confined aquifer in which the water level in a well completed within the aquifer rises above the top of the aquifer.

Confined Aquifer - An aquifer confined between two low permeability layers (aquitards).

Equipotential Line - A contour line on the potentiometric surface or water table showing uniform hydraulic head levels. Equipotential lines on the water table are also called water table contour lines.

Flow Line - A line indicating the direction of groundwater movement within the saturated zone. Flow lines are drawn perpendicular to equipotential lines.

Flow Net - A diagram of groundwater flow, showing flow lines and equipotential lines.

Piezometric Head - The height to which water will rise in a cased well.

Potentiometric Surface - A surface which is defined by the levels to which water will rise in cased wells which are screened in a specified zone of an unconfined aquifer or in a confined aquifer.

Unconfined (water table) Aquifer - An aquifer in which the water table forms the upper boundary.

Water Table - A surface in an aquifer where groundwater pressure is equal to atmospheric pressure and below which all strata are saturated with water.

9.0 ATTACHMENTS

None

Earth Resources Technology, Inc. Standard Operating Procedure **BOREHOLE LOGGING**			
Effective Date: **01/22/09**	Version: **001**	SOP#: **ERT SOP-07-3**	
Approvals			
Mike Dorman Program Manager		R. Russell Ashley, C.P.G. Field QA Manager	
Signature	1/23/09 Date	Signature	1/22/09 Date

1.0 PURPOSE

The purpose of this procedure is to define the minimum requirements for field logging of boreholes and environmental samples. Consequently, the major objectives of this plan are to provide a uniform set of guidelines that will aid in developing consistency among media descriptions and sample techniques. The importance of accurate, complete, clear and concise logs cannot be overemphasized.

2.0 SCOPE

This procedure applies to descriptions of the standard techniques used for logging boreholes and logging soil/rock samples.

3.0 REQUIREMENTS

Careful field documentation and sample description is necessary to ensure that logging is done in a consistent manner.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for ensuring that field personnel have been trained in the use of this procedure ensuring consistency in logging methodology between Field Personnel and compiling the boring logs for inclusion into a field report.

4.2 Field Team Leader

The Field Manager/Field Team Leader is responsible for ensuring that borehole logging methods detailed in this procedure are followed and that consistency between descriptive nomenclatures from various logs for a site are accurate. The Field team Leader is responsible for reviewing logs for completeness and accuracy.

4.3 Field Team

The Field Team is responsible for accurately recording (logging) pertinent information regarding geologic materials encountered during the boring operations.

5.0 EQUIPMENT

The following is a list of required and optional equipment necessary for borehole logging.

5.1 Required Equipment

1. Clipboard
2. Soil Boring Forms (Attachment 4)
3. Photoionization Detector (PID) with calibration gas
4. Field Logbook
5. Folding Rule or Tape Measure (calibrated to 0.01 foot)

6. Color Chart
7. Water Level Indicator
8. Copy of Drilling Contract
9. Waterproof Permanent Marking Pen
10. Sample Jars or Bags

5.2 Optional Equipment

1. Hand Lens X10
2. Brunton Compass
3. Pocket Penetrometer
4. Equipment Pouch
5. Flagging Tape
6. Cooler and Water Bottles
7. Flashlight
8. Rock Hammer
9. Acid Bottle
10. Digital Camera

6.0 PROCEDURE

6.1 Recording Data

Borehole information that is required for inclusion in the Field Logbook is detailed in ERT SOP-01-2. In addition to information recorded in the Field Logbook, it is required that soil logging data be recorded on a pre-printed boring-specific log (Attachment 4). This activity is done to provide a clear and concise record of borehole lithology.

6.2 Information to be Gathered During Borehole Logging

Frequency and detail of borehole logging will be detailed in the Sample and Analysis Plan. When conducting discreet sampling, the first recorded data for each sample should be the recovery of soil from the core of split spoon. Recovery will be recorded in inches using a tape measure. When measuring recover, attention must be made to disregard “sluff” that typically precedes the actually sample volume. Secondly, a photoionization detector (PID) reading should be taken from a minimum of three locations long the core sample and recorded in the field logbook. Finally, a description of the geologic unit(s) should be detailed including moisture, constituents, odor, color, and other such descriptors. Careful attention to detail should be made in regards to changes in homogeneity of a sample. Features potentially related to impacts should also be noted such as odor, staining and PID readings. The description of lithologic samples should include color, material description, moisture content, and consistency of the sample or unit. These characteristics should be described according to guidelines given in the attachments. Soil sampling will be performed in accordance with ERT SOP-06-3 and the sampling intervals noted on the log form. Samples screened with a PID are to have the concentrations noted in the appropriate section of the log form, corresponding to the sample interval.

6.3 Logging Guidelines

For accuracy and consistency, boring log descriptions should generally be completed in the following order. Refer to the listed attachments for guidance.

1. Color. Color should be described using a Munsell color chart, and the colors listed in the chart only. If the colors in the sample are variable, adjectives such as "mottled" or

"banded" may be used as appropriate (See Attachment 2). In the written description, the color should be given first with the first letter capitalize.

2. Material Description. Material description is the written description of the material being sampled or observed. In the written description the color (capitalized) is followed by the soil components from most prevalent (capitalized) to least. Refer to Attachment 2 for guidance on soil description procedures.

For example – Brown coarse to fine sand, little Clayey Silt, some (-) medium to fine (+) Gravel.

3. Description of mineralogy may be included in the material description and should be as simple as possible and, above all, accurate. Relatively common mineralogic descriptions may be used as adjectives:
   - Arkosic
   - Calcareous
   - Feldspathic
   - Glauconitic
   - Micaceous
4. Moisture Content. If the drilling method permits, the moisture content of the sample (moist, damp or wet) should be noted. Refer to the attachments for guidance.

5. Consistency. Consistency is the density or strength of the soil. See Attachment 3.

6. USCS Classification. See Attachment 1.

7.0 REFERENCES

The following documents were utilized in the creation of this Standard Operation Procedure:

Compton, R. R., 1985. *Manual of Field Geology*, John Wiley and Sons, Inc., New York.

Folk, R. L., 1968. *Petrology of Sedimentary Rocks*, Hemphills Bookstore, Austin, Texas, **p. 170.**

Lewis, D. W., 1984. *Practical Sedimentology*, Van Nostrand Reinhold Company, Inc., New York, New York.

Pettijohn, F. J., 1975. *Sedimentary Rocks*, Harper & Row, New York.

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 DEFINITIONS

This section provides information that is commonly used in borehole sample descriptions (also see Section 9.0 Attachments).

Bedding - Existence of beds or layers (strata), laminae, or other tabular and essentially horizontal units.

Cohesive - Having the capacity to stick together or adhere together. In effect, the cohesion of soil is that part of its shear strength which does not depend on interparticle friction.

Color - Described using a Munsell color chart, and only colors listed in that chart should be used. If the colors in the sample are variable, adjectives such as "mottled" or "banded" may be used as appropriate.

Conchoidal - Shell-like surface form produced by fracture of a brittle material.

Consistency - Density or strength of the soil, and is a primary factor in engineering investigations (see Attachment 9.3).

Fabric - Orientation of the particles composing a soil or rock.

Friable - Easily crumbled.

Grading - Degree of mixing size classes in a sedimentary material. Well graded implies uniform distribution from coarse to fine; poorly graded implies lack of uniformity in size or lack of continuous distribution (also see sorting).

Grain Size - Size of particles within a rock or a soil sample (see Attachment 2).

Moisture - Degree of wetness of a soil, i.e. moist, damp and wet.

Plasticity - Property of a material which enables it to undergo permanent deformation without appreciable volume change or elastic rebound, and without rupture.

Slickensides - Polished and striated (scratched) surface that results from friction along a fault plane. Apparent slickensides can sometimes be created during the drilling process.

Soil Classification - See Attachment 1.

Texture - Geometric aspects of the component particles of a soil or rock, including size, shape, and arrangement.

9.0 ATTACHMENTS

1 USCS Classification
2 Definition of Soil Components
3 Field Observations for Determining Soil Consistency
4 Boring Log Forms

Attachment 1

USCS Classification

MAJOR DIVISIONS			LETTER SYMBOL	TYPICAL DESCRIPTION
COARSE GRAIN SOILS MORE THAN 50% OF MATERIAL IS LARGER THAN NO.200 SIEVE SIZE	GRAVEL AND GRAVELY SOILS MORE THAN 50% OF COARSE RETAINED IN NO.4 SIEVE	CLEAN GRAVELS LITLE OR NO FINES	GW	WELL GRADED GRAVELS, GRAVEL SAND MIXTURES, LITTLE OR NO FINES
			GP	POORLY GRADED GRAVELS, GRAVEL SAND MIXTURES, LITTLE OR NO FINES
		GRAVELS WITH FINES APPRECIABLE AMOUNT OF FINES	GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES
			GC	CLAYEY GRAVELS, GRAVEL-SAND-CLAY MIXTURES
	SAND AND SANDY SOILS MORETHAN 50% OF COARSE FRACTION PASSING NO.4 SIEVE	CLEAN SAND LITTLE OR NO FINES	SW	WELL GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES
			SP	POORLY GRADED SANDS, GRAVELLY SAND, LITTLE OR NO FINES
		SANDS WITH FINES APPRECIABLE AMOUNT OF FINES	SM	SILTY-SANDS, SAND-SILT MIXTURES
			SC	CLAYEY SANDS, SAND-CLAY MIXTURES
FINE GRAINED SOILS MORE THAN 50% OF MATERIAL IS SMALLER THAN NO.200 SIEVE SIZE	SILTS AND CLAYS LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS OR CLAYEY SILTS WITH SLIGHT PLASTICITY
			CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
			OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
	SILTS AND CLAYS LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SAND OR SILTY SOILS
			CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS
			OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICTY, ORGANIC SILTS
HIGHLY ORGANIC			PT	PEATS, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENT

Attachment 2

Definition of Soil Components

GRANULAR MATERIAL	SIZE DESCRIPTION	SIZE	GRANULAR MATERIAL	SMALLEST DIAMETER OF ROLLED THREADS
BOULDERS		> 23 cm (9 in)	Silt	none
COBBLES		7.6 cm (3 in) - 23cm (9 in)	Clayey SILT	6 mm (1/4 in)
GRAVEL	COARSE	25 mm (1 in) - 76 mm (3 in)	SILT and CLAY	3mm (1/8 in)
	MEDIUM	9.5 mm (0.375 in) - 25 mm (1 in)	CLAY and SILT	1.5 mm (1/16 in)
	FINE	2 mm (0.08 in) - 9.5 mm (0.375 in)	Silty Clay	0.8 mm (1/32 in)
SAND	COARSE	0.59 mm - 2 mm	CLAY	0.4 mm (1/64 in)
	MEDIUM	0.25 mm - 0.59 mm		
	FINE	0.074 mm - 0.25 mm		

Attachment 3

Field Observations for Determining Soil Consistency

Clay

Clay Consistancy	Thumb Penetration	Average SPT N Blows/ft over 6-18" interval of 24" split spoon
VERY SOFT	Easily penetrated several inches by thumb. Exudes between thumb and fingers when squeezed in hand.	< 2
SOFT	Easily penetrated one inch by thumb. Molded by light finger pressure.	2 – 4
MEDIUM STIFF	Can be penetrated over ¼ inch by thumb with moderate effort. Molded by strong finger pressure.	4 – 8
STIFF	Indented about ¼ inch by thumb but penetrated only with great effort.	8 – 15
VERY STIFF	Readily indented by thumbnail	15 – 30
HARD	Indented with difficulty by thumbnail.	> 30

Sand

SOIL TYPE	Average SPT N Blows/ft over 6-18" interval of 24" split spoon	RELATIVE DENSITY %	FIELD TEST
VERY LOOSE SAND	4	0 - 15	Easily penetrated with ½" reinforcing rod pushed by hand.
LOOSE SAND	4 – 10	15 – 35	Easily penetrated with 1/2" reinforcing rod pushed by hand.
MEDIUM DENSE SAND	10 – 30	35 – 65	Penetrated a foot with ½" reinforcing rod driven by 5-lb hammer.
DENSE SAND	30 – 50	65 – 85	Penetrated a foot with ½" reinforcing rod driven by 5-lb hammer.
VERY DENSE SAND	<50	85 - 100	Penetrated only a few inches with ½" reinforcing rod driven by 5-lb hammer.

Attachment 4

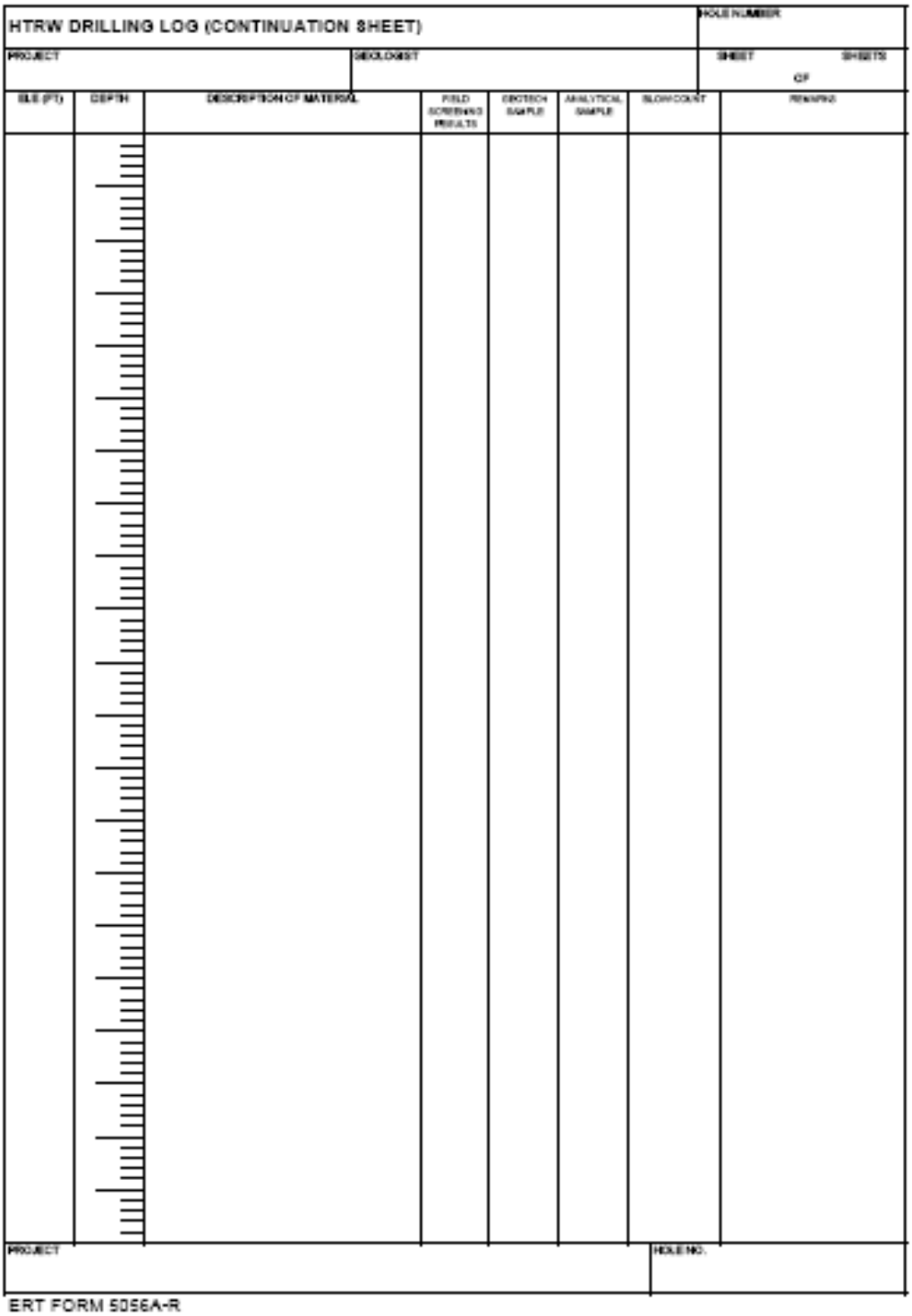
Boring Log Forms

NOTE: Earth Resources Technology currently utilizes two different formats of boring log forms. The determination as to which form will be utilized is based on the clientele of the contract being executed.

ERT Form 5056 is modeled after the United States Corp of Engineers ENG Form 5056-R and ENG Form 5056A-R and is used when operating under USACE contract.

HTRW DRILLING LOG							
	DISTRICT					HOLE NUMBER	
1. COMPANY NAME	2. DRILLING CONTRACTOR					SHEET OF	SHEETS
3. PROJECT			4. LOCATION				
5. NAME OF DRILLER			6. MANUFACTURS DESIGNATION OF DRILL				
7. SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT			8. HOLE LOCATION				
			9. SURFACE LOCATION				
			10. DATE SAMPLED	11. DATE COMPLETED			
12. OVERBURDEN THICKNESS			15. DEPTH GROUND WATER ENCOUNTERED				
13. DEPTH DRILLED INTO ROCK			16. DEPTH TO WATER AND ELAPSED TIME SINCE DRILLING COMPLETED				
14. TOTAL DEPTH OF HOLE			17. OTHER WATER LEVEL MEASURMENTS SPECIFY				
18 GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES				
20. SAMPLES FOR CHEM ANAL	VOC	METALS	OTHERS (SPECIFY)	OTHERS (SPECIFY)	OTHERS (SPECIFY)	21. TOTAL CORE RECOVERY	
22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHERS (SPECIFY)	23. GEOLOGIST			
LOCATION SKETCH / COMMENTS			SCALE:				
PROJECT			HOLE NO.				

ERT FORM 5056-R



HTRW DRILLING LOG (CONTINUATION SHEET)

HOLE NUMBER

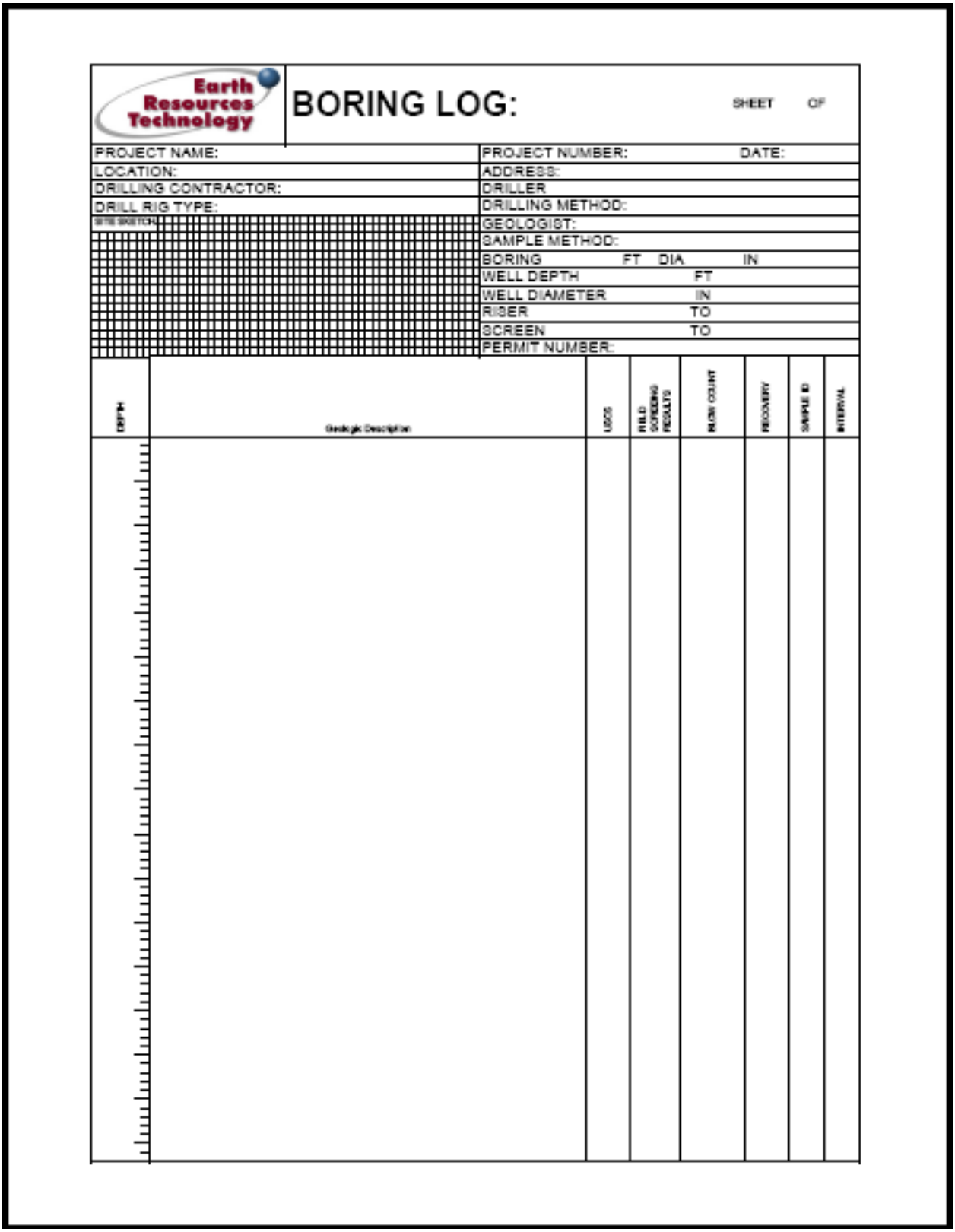
PROJECT | GEOLOGIST | SHEET | SHEETS OF

ELE (FT)	DEPTH	DESCRIPTION OF MATERIAL	FIELD SCREENING RESULTS	SAMPLE	ANALYTICAL SAMPLE	BLOW COUNT	REMARKS

PROJECT | HOLE NO.

ERT FORM 5056A-R

For Non-USACE clientele ERT utilizes *ERT Well Logging Form V4*.



BORING LOG: SHEET OF

PROJECT NAME:	PROJECT NUMBER: DATE:
LOCATION:	ADDRESS:
DRILLING CONTRACTOR:	DRILLER
DRILL RIG TYPE:	DRILLING METHOD:
SITE SKETCH:	GEOLOGIST:
	SAMPLE METHOD:
	BORING FT DIA IN
	WELL DEPTH FT
	WELL DIAMETER IN
	RISER TO
	SCREEN TO
	PERMIT NUMBER:

DEPTH	Geologic Description	USCS	FIELD SCREENING RESULTS	BLOW COUNT	RECOVERY	SAMPLE ID	INTERVAL

ATTACHMENT 4
CT Laboratories Analytical Method SOPs

CT Laboratories
Baraboo Laboratory Divison

Title: **Graphite Furnace Atomic Absorption**

SOP Number: **6110B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

A large number of metals in solution may be determined by graphite furnace atomic absorption spectroscopy. This procedure is used for low-level metals analysis of drinking water, groundwater, wastewater, soil, and sludge. SDWA samples and clean groundwater samples, properly preserved, with <1 NTU of turbidity may be analyzed directly; otherwise, sample digestion is required. All other sample matrices require digestion prior to analysis.

2.0 METHOD SUMMARY

A representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a hollow cathode lamp specific to the analyte of interest is passed through the center of the graphite tube. The intensity of the transmitted radiation decreases in proportion to the amount of the analyte specific ground-state atoms present in the graphite tube upon atomization. A monochromator isolates the characteristic radiation from the hollow cathode lamp, and a photosensitive device measures the transmitted radiation. The SIMAA can measure up to 4 elements simultaneously. The SIMAA employs the Zeeman background correction technique. Matrix interferences from organic and inorganic constituents are minimized when the appropriate background correction technique is used in conjunction with the appropriate matrix modifiers.

3.0 DEFINITIONS

3.1 Reagent Blank - A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.2 Calibration Standards - A series of known standard solutions, which may include the reagent blank, used for calibration of the instrument within the measurable linear range. Calibration standards should contain, in correct proportion, all reagents required by the method. A minimum of 4 calibration points plus a reagent blank are used for calibration. Acceptance of the calibration requires a correlation coefficient of 0.995 or better. No samples will be analyzed without acceptable calibration. AA calibrations are performed daily.

3.3 Calibration Verification Standards-Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. Note that the ICV must be from a different source than that of the calibration standards and the CCV, and that limits for ICV are tighter than those for CCV.

3.4 (ICB/CCB (Calibration Blanks- Initial and Continuing) - A reagent blank solution, which is analyzed immediately following the calibration standards (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end

of a run to check for drifts in calibration, or possible analyte carry-over.. Warning criteria include that the absolute value be less than or equal to the MDL for a given analyte. Control criteria consist of the absolute value being less than or equal to 2 times the MDL for a given analyte. If this range is exceeded, a new calibration will be necessary.

3.5 LCS (Laboratory Control Sample)- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the calibration at the start of an analytical run, and is analyzed immediately after the initial CB (ICB).

3.6 PB or MB (Prep Blank/ Method Blank) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch.

3.7 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every 20 samples of a given matrix per day for 7000 series and CLP and every 10 samples of a given matrix per day for 200 series. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To be considered acceptable, MSD's must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD and may be used as acceptance criteria for duplicate analysis.

3.8 Detection Limit (DL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

4.0 HEALTH AND SAFETY

4.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

The SIMAA employs high magnetic fields during operation. People with pacemakers should be kept away from the instrument area.

6.0 INTERFERENCES

6.1 Direct aspiration

6.1.1 The most troublesome type of interference in atomic absorption spectrophotometry is usually termed "chemical" and is caused by lack of absorption of atoms bound in molecular

combination in the flame.This phenomenon can occur when the flame is not sufficiently hot to dissociate the molecule, as in the case of phosphate interference with magnesium, or when the dissociated atom is immediately oxidized to a compound that will not dissociate further at the temperature of the flame.

6.1.2 Chemical interferences may also be eliminated by separating the metal from the interfering material. Although complexing agents are employed primarily to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

6.1.3 The presence of high dissolved solids in the sample may result in an interference from nonatomic absorbance such as light scattering.

6.1.4 Spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal. Interference can also occur when resonant energy from another element in a multielement lamp, or from a metal impurity in the lamp cathode, falls within the bandpass of the slit setting when that other metal is present in the sample. This type of interference may sometimes be reduced by narrowing the slit width.

6.1.5 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate All metals are not equally stable in the digestate, especially if it contains only nitric acid, not nitric acid and hydrochloric acid. The digestate should be analyzed as soon as possible, with preference given to Sn, Sb, Mo, Ba, and Ag.

6.2 Furnace procedure

6.2.1 Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition
of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:
1) Successively dilute and reanalyze the samples to eliminate interferences.
2) Modify the sample matrix either to remove interferences or to stabilize the analyte. Examples are the addition of ammonium nitrate to remove alkali chlorides and the addition of ammonium

phosphate to retain cadmium. The mixing of hydrogen with the inert purge gas has also been used to suppress chemical interference. The hydrogen acts as a reducing agent and aids in molecular dissociation.
3) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

6.2.2 Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. When this occurs, use either background correction or choose an alternate wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.

6.2.3 Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

6.2.4 Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

6.2.5 Cross-contamination and contamination of the sample can be major sources of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If suspected, they should be acid soaked with 1:5 nitric acid and rinsed thoroughly with tap and reagent water. Special attention should be given to reagent blanks in both analysis and in the correction of analytical results. Lastly, pyrolytic graphite, because of the production process and handling, can become contaminated. As many as five to ten high-temperature burns may be required to clean the tube before use.

7.0 PERSONNEL QUALIFICATIONS

Personnel operating the graphite furnace should have background knowledge of the scientific principles used during this application. All operators should perform an initial demonstration of capability (IDC) prior to analyzing any samples. It is preferable for the operator to have at least two semesters of college chemistry.

8.0 APPARATUS AND MATERIALS

8.1 Apparatus and Materials

8.1.1 Perkin-Elmer SIMAA simultaneous spectrophotometer with autosampler.

8.1.2 Graphite furnace tubes: THGA platform tubes.

8.1.3 Sample carousel standard cups.

8.1.4 Plastic autosampler vials.

8.1.5 Electrodeless Discharge Lamps (EDL).

8.1.6 Argon: high purity.

8.1.7 Class `A' volumetric flasks and pipettes.
8.1.8 Eppendorf pipettes.

8.2 REAGENTS

8.2.1 Alternate source multi-element stock standard, purchased Inorganic Ventures, # CTI-GFCAL-1 or equivalent.
10 ug/mL Sb, As, Cr, Pb, Se, Tl
1 ug/mL Cd, Ag.

8.2.2 Multi-element stock standard, purchased Inorganic Ventures, #CTI-SPK-1 or equivalent
100 ug/mL Sb, As, Cr, Pb, Se Tl
15 ug/mL Ag
5 ug/mL Cd

8.2.3 Single-element standard-1000 ug/mL Sn

8.2.4 Single-element standard- purchased 1000 ug/mL Sn- source alternate to 8.2.3

8.2.5 Concentrated nitric acid (HNO_3): Trace metals grade.

8.2.6 Type II water: 18-megohm Deionized water.

8.2.7 1% Palladium solution, purchased

8.2.8 1% Magnesium Nitrate solution, purchased

8.2.9 Citric Acid (powdered).

8.2.10 Ammonium Dihydrogen Phosphate (powdered).

8.2.11 Standard preparation:

8.2.11.1 Base Standard: To a 200-mL volumetric flask, add 0.5 mL conc. HNO3 and 0.1mL Inorganic Ventures CTI-SPK-1 or equivalent (8.2.2). Bring to volume with Milli-Q water and invert several times to mix. Prepare annually or as needed. Concentration: 50 ug/L Sb, As, Pb, Se, Tl and 7.5 ug/L Ag

8.2.11.2 Base Standard: To a 200-mL volumetric flask, add 0.5 mL conc. HNO3 and 0.02mL 1000mg/L Sn (8.2.3). Dilute to volume with Milli-Q water and invert several times to mix. Prepare annually or as needed. Concentration: 100 ug/L Sn

8.2.11.3 ICV/CCV-Check Standard: To a 200-mL volumetric flask, add 0.5 mL conc. HNO3 and 0.5 mL of Inorganic Ventures CTI-GFCAL-1 (8.2.1) or equivalent. Dilute to volume with Milli-Q water. Prepare annually or as needed. Concentrations: 25 ug/L Sb, As, Pb, Se, Tl and 2.5 ug/L Ag

8.2.11.4 ICV/CCV-Check Standard: To a 200-mL volumetric flask, add 0.5 mL conc. HNO3 and 0.01 mL of 1000 mg/L Sn (8.2.4). Dilute to volume with Milli-Q water and invert several times to mix . Prepare fresh annually or as needed. Concentrations: 50 ug/L Sn

8.2.12 Matrix modifier preparation:

8.2.12.1 Palladium Nitrate 0.05%: To a 200-mL volumetric flask, add 100 ml DI H2O. Add 4 g. citric acid, 10 ml 1% Palladium Nitrate solution, and 1 mL 1% Magnesium Nitrate solution. Dilute to volume with DI H2O. Prepare fresh every 6 months. To be used for the analysis of Sb, Tl, As, Se, Sn.

8.2.12.2 Ammonium Phosphate 0.5%: To a 200-mL volumetric flask, add 1.0 g. Ammonium Dihydrogen Phosphate. Dilute to volume with DI H2O. Prepare fresh every month. To be used for the analysis of Cd and Pb.

9.0 INSTRUMENT CALIBRATION

See section 11.0 for calibration details.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

	Aqueous	Solids
Preservative:	pH <2 with HNO_3	cool 4 ^{o}C
Hold Time:	180 days	180 days

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 SIMAA

11.1.1 Turn spectrophotometer on. All necessary power up functions is carried out automatically by the instrument. On the computer screen double click on the SIMAA icon. A sequence of windows will open up. Call up the correct method from the method library. See Appendix A for recommended calibration concentrations.Note: All methods are stored in the method library section of the software. For individual furnace temperature profiles consult the stored program.

11.1.2 Now that the method is loaded, you will install all the EDL or hollow cathode lamps needed to carry out the analysis. Allow a full hour warm-up for EDL and 30 minutes for HCL. After warm-up, align and optimize each lamp with the adjustment wheels on the lamp cassette. Record the lamp reading in the lamp logbook.

11.1.3 Alignment of the furnace head is not necessary unless the contacts have been replaced since the last time the instrument was operated. If it does need alignment consult the operations manual.

11.1.4 Check alignment of the autosampler tip with respect to the graphite tube and the sample cups. See operations manual.

11.1.5 Inspect graphite tube. If worn replace before analysis. If tube is replaced, press “condition tube” on furnace control page.

11.1.6 Place modifier or modifiers, base std, blank, and rinse blank into the positions on the autosampler designated in the program.

11.1.7 Create a new sample info file. Input all sample ID’s including LCS duplicates, etc. Designate which samples to auto-spike if there are

any. Save file with element symbol and today's date Ex) Pb030999. Print Autosampler loading list or sample info file.

11.1.8 Load autosampler, and on analysis page click on ANALYZE ALL-CALIBRATION and SAMPLES. Record the samples and sample info file in the SIMAA sample logbook.

11.1.9 After instrument has calibrated, record the characteristic mass of the element in the SIMAA run logbook. If chacteristic mass is not within 50-200% of the characteristic mass obtained during the MDL study, stop analysis and correct problem.

11.1.10 For Data Capture

11.1.10.1 When instrument is "idle" after running samples, open reformat data in AAWinLab by clicking on file and then reformat data

11.1.10.2 Click on open design and choose data capture

11.1.10.3 Choose the result data set by clicking on browse and choose the appropriate data set.

11.1.10.4 Name the file to be reformatted as the LIMS analytical run number

11.1.10.5 Click on save results.

11.1.10.6 Close reformat data section

12.0 INSTRUMENT MAINTENANCE AND TROUBLESHOOTING

12.1 Refer to Perkin Elmer operator manual for further troubleshooting information.

12.2 Daily Maintenance

12.2.1 Check and, if need be, replace the graphite tube.

12.2.2 Check and, if need be, clean or replace contact cylinders

12.2.3 Check sampler tip in tube. Check sampler tip alignment

12.2.4 Flush sampler

12.2.5 Change fume extractor water

12.2.6 Inspect fume extractor filter and replace if dirty

12.2.7 Check rinse reservoir and fill if needed

12.2.8 Empty waste reservoir if needed

12.2.9 Check argon gas tank to ensure there is suffient gas and that it is at the appropriate pressure.

13.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

Sample Calculations:

Liquid Concentration (ug/L) = A x C

Solid Concentrations (mg/kg) = $\frac{A \times B \times C}{D \times E}$,

where A = instrument reading for sample (ug/L)

B = total volume of digestion (L)
C= analyst dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary

$$\textit{Spike Recovery (\%)} = \frac{\text{(Spiked sample concentration – Sample concentration) x 100}}{\text{(Spike amount)}}$$

$$\textit{\%RSD} = \frac{\text{(MS – MSD) x 100}}{\text{(MS + MSD)/2}},$$

where MS = Matrix spike concentration
MSD = Matrix spike duplicate concentration

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS
14.2 Computer with Perkin Elmer AAWinLab

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.
15.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
15.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.
15.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 All calibrations are done within the linear range of the instrument. The acceptance criteria for linearity is a correlation coefficient of 0.995 or better. To ensure linearity all calibrations are made up of a blank and 5 standards. The highest standard used for each calibration is within the linear range for that element or combination of elements in the case of the SIMAA. Any sample measuring above the concentration of the highest standard is diluted until it is within range then reanalyzed.
16.2 Non-CLP (7000A, 200.9Modified):
16.2.1 ICV (initial calibration verification): Following instrument calibration, begin the analysis with the 'QC Standard'. ICV should be from a different source than the calibration standards. If recovery exceeds the following limits, terminate the run and correct the problem before proceeding.

SW846-method 7000A	SDWA- method 200.9Mod
90-100%	95-105%

ICB (initial calibration blank): Following the ICV, analyze a blank. The absolute value of the ICB must be below the MDL or reporting limit for the method or sample results must be less than MDL or greater than 20 times the blank for SDWA and below two times the MDL or reporting limit for the method or sample results must be less than MDL or greater than 20 times the blank for SW846.. If the result exceeds this, terminate the analysis and correct the problem before proceeding or qualify data.

16.2.2 LCS (laboratory control sample): Analyze an alternate source standard that has gone through the same preparation process as the samples. Use in-house control limits if available or use the default limits of 80-120% recovery for SW-846 and 90-110% for SDWA. If the recovery exceeds the limits, terminate the analysis and correct the problem before proceeding.

16.2.3 CCV (continuing calibration verification): Same standard used for the calibration standanrds but analyzed after every ten samples followed by a CCB. Must be also be run at the end of the analytical run. If the recovery exceeds the following limits, you must recalibrate and reanalyze all samples back to the last acceptable CCV or ICV.

SW846-method 7000A	SDWA-method 200.9Mod
80-120%	90-110%

16.2.4 CCB (continuing calibration blank): Analyze a blank following every CCV. The absolute value of the CCB must be below the MDL or reporting limit for the method or sample results must be less than MDL or greater than 20 times the blank for SDWA and below two times the MDL or reporting limit for the method or greater than 20 times the blank for SW846. If the result exceeds this, reanalyze all samples back to the last acceptable CCB or qualify data.

16.2.5 MS/MSD (Matrix spike/Matrix spike Duplicate): A MS/MSD is required every analytical run at a frequency of 5% per digestion batch for 7000 series or at a frequency of 10% per digestion batch for 200 series. For digested samples, appropriate samples are designated and spiked at the time of the digestion. For non-digested samples, a bench spike can be prepared with the Varian. The SIMAA will automatically dispense a predetermined amount into the designated samples. See operations manual.

16.2.5.1 The results from the MS-MSD are reviewed for both accuracy limits and precision limits. Acceptance criteria categorized by matrix found in the lab Quality Control Limits Sheet issued quarterly by Quality Assurance. These limits are applicable when the spike level is > 25% of the original analyte level in the sample (EPA 200.9Modified

and SW7000A). Failure to meet this criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To help determine the reason for the failure, a post-digestion spike is performed on failed samples to ascertain the role of the digestate matrix on the spike failure.

16.2.6 PDS (Post Digestion Spike) - When a matrix spike falls outside of the 25% acceptance range, a post digestion spike is used to determine if the sample digestate matrix is interfering with the analysis of the analyte. The sample is spiked at a level consistent with the matrix spike. Acceptable recovery is within 15% of the spike true value, indicating that the sample digestate matrix is not interfering with analysis. If the recovery is outside of that range, than the digestate matrix is causing interference with analysis and the PDS may be used as the single addition method of standard additions (MSA) to correct the sample result and to recalculate the MDL, as appropriate. **Note: For ACOE (LCG) samples, a PDS will be performed at a rate of one per batch per matrix.**

16.2.7 Dilution Analysis – An SW846 sample digestate is diluted x5 with method blank solution and analyzed. The diluted digestate result and the undiluted digestate result should agree with less than 10% RPD, or a matrix effect is suspected. This is performed on one the MS/MSD sample for each batch of solid samples. **Note: For ACOE samples, a SD will be performed at a rate of one per batch per matrix.**

16.2.8 PB (preparation blank): From the digestion batch. If the result exceeds the CRDL and sample results are greater than the CRDL and less than 20 times the PB, re-digest all samples associated with the PB.

16.3 CLP-like Protocol

16.3.1 ICV (initial calibration verification): Following instrument calibration, begin the analysis with the 'QC Standard'. If the recovery exceeds the following limits, terminate the run and correct the problem before proceeding. ICV limit: 90-110%

16.3.2 ICB (initial calibration blank): Following the ICV, analyze a blank. The absolute value of the result for the ICB must be below the LOD for the method or below the level set by the client QAPP (1/2 the MRL for ACOE work). If the result exceeds this, terminate the analysis and correct the problem before proceeding.

16.3.3 CRDL/MRL standard: Analyze a standard at the CRDL or at a level set by the client. Limits are established by the client (70-130% for ACOE work)

16.3.4 PB (preparation blank): From the digestion batch. If the result exceeds the CRDL (1/2 the CRDL for ACOE work) and sample results are less than 20 times the PB and greater than the CRDL, re-digest all samples associated with the PB.

16.3.5 LCS (laboratory control sample): From the digestion batch. Analyze an alternate source reference sample. Use in-house control limits or client limits from QAPP if available or use the default limits of 80-120% recovery. If the recovery exceeds the limits, terminate the analysis and correct the problem before proceeding.

16.3.6 CCV (continuing calibration verification): Analyze the CCV following every ten samples, and at the end of the analysis. If the recovery exceeds the following limit, recalibrate and reanalyze all samples back to the last acceptable CCV. CCV Limit: 80-120% or as set by client QAPP.

16.3.7 CCB (continuing calibration blank): Analyze a blank following every CCV. The absolute value of the result for the CCB must be below the LOD for the method (less than ½ the MRL for ACOE work) or sample results bracketed by the CCB must be greater than 20 times the CCB or less than the LOD (or less than MRL for ACOE work). If the CCB result exceeds this, reanalyze all samples back to the last acceptable CCB.

16.3.8 Matrix Spike (MS): A matrix spike is required every for every sample delivery group of 20 samples or less or as required by the client QAPP. The matrix spike shall be prepared at the time of digestion. Use in-house conrol limits or client limits from QAPP if available or use the default limits of 75-125% recovery.

16.3.9 Duplicate: A duplicate is required for every sample delivery group of 20 samples or less or as required by client QAPP and is prepared at the time of digestion . For results exceeding five times the CRDL, use in-house control limits or client limits from QAPP if available or use the default limit of 20% RPD. For results that are less than five times the CRDL, the default control limit is +/- CRDL. If the recovery is outside this, see appropriate QAPP or ACOE "LCG" document for corrective action.

16.4 Any results that fall outside the limits established are flagged and noted in the Metals Data Review form attached to the data package.

16.5 For each run a "QC Bench Sheet" is completed, summarizing accuracy, precision, spike preparation, and calibration standard references. Data from these sheets are used to create quarterly control limits.

16.6 Data review is documented on the analysis data review checklist.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*, EPA, 1986, SW-846, 3rd Edition, Rev 1 July 1992 Method 7000

17.2 *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010. Rev 2.2 May 1994. Method 200.9.

17.3 USEPA Contract Laboratory Program. Statement of Work for Inorganic Analysis. ILM03.0.

17.4 Perkin Elmer Simultaneous Multielement AA Spectrophotometer Techniques- Operation and methods manuals

18.0 Tables

Pre-Digestion Matrix Spike Prep for (GFAA)

Element	Spike Amt. mL	Spike Soln.	Stock Conc. ug/L	Final Vol. mL	Expected Conc. Ug/L
Antimony-GFAA	1.0	**A**	1000.0	50.0	20.0
Arsenic-GFAA	1.0	**A**	1000.0	50.0	20.0
Chromium-GFAA	1.0	**A**	1000.0	50.0	20.0
Lead-GFAA	1.0	**A**	1000.0	50.0	20.0
Selenium-GFAA	1.0	**A**	1000.0	50.0	20.0
Thallium-GFAA	1.0	**A**	1000.0	50.0	20.0
Silver-GFAA	1.0	**A**	150.0	50.0	3.00
Cadmium-GFAA	1.0	**A**	50.0	50.0	1.00
Tin-GFAA	0.1	**B**	10,000	50.0	20.0

Standard Source		
Supplier	**Dilution**	**Solution**
Inorg Ventures.-SPK1	1/100	**A**
VWR Sn std.	1/100	**B**

Analytical (non-digested) Spike Analysis (GFAA)

Element	Spike Amt. Uls	Spike Solution	Stock Conc. ug/L	Final Volume UL	Expected Conc. ug/L
Antimony-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Arsenic-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Chromium-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Lead-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Selenium-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Thallium-GFAA	10.0	**GF-CAL**	50.0	25.0	20.0
Silver-GFAA	10.0	**GF-CAL**	5.00	25.0	2.00
Cadmium-GFAA	10.0	**GF-CAL**	5.00	25.0	2.00
Tin-GFAA	10.0	**JT Baker Sn**	50.0	25.0	20.0

Standard Source		
Supplier	**Expires**	**Prep Info**
Inorg Vent.-GFCAL	11/1/2000	1/200 dil. of concentrate
JT Baker Sn Std.	4/2000	1/20000 of concentrate

Standard Quality Control Requirements and Corrective Action Guidelines

QC Type	Frequency	Conc. Level	*Acceptance Criteria*	*Corrective Action*
ICV	1 per calibration	20 ug/L	90-110%	Terminate run. Correct the problem before proceeding
ICB	Immediately after ICV	<MDL	SDWA: < MDL SW846: < MDL CLP:ABS<1/2 MRL or as stated in QAPP	Flag: Analyte detected in ICB or recalibrate
MB	1 per batch of 20 samples	<MDL	SDWA: < MDL SW846: < MDL CLP:ABS<1/2 MRL or as stated in QAPP	Flag: Analyte detected in Method Blank or reprep
LCS	1 per batch of 20 samples	mid cal. Range	In-house limits or, default 80-120% SDWA; 90-110%	Terminate analysis: correct problem before proceeding.
CCV	1 after every 10th sample	mid cal range	80-120%	Recalibrate and reanalyze all samples back to the last acceptable CCV or ICV
CCB	Immediately following each CCV	<MDL	SDWA: < MDL SW846: < MDL CLP:ABS<1/2 MRL or as stated in QAPP	Reanalyze all samples back to the last acceptable CCB or ICB or flag analyste detected
MS-MSD	5% (1 in 20) of samples per batch per matrix	See attached spike chart	In-house limits or, default of 75-125 when spike level is >25% of original analyte level	Perform PDS
Serial Dilution Analysis	1 per digestion batch per matrix	5 fold dilution of chosen sample	RPD within 10% of value of diluted and undiluted sample, but only if sample conc. >/= 50 X's LOD	Flag data only if sample conc. Is within range (>50X's LOD)

Post Digestion Spike (PDS)	Upon failure of MS and/or 1 per batch for ACOE work	Same level as MS	85-115%	Flag: Matrix interference or Method of Std. Additions

Appendix A
Recommend Calibration Curve Concentrations

Element	Concentrations (ug/L)
Antimony	0, 4, 10, 20, 30, 40
Arsenic	0, 4, 10, 20, 40, 50
Lead	0, 4, 10, 20, 40, 50
Selenium	0, 4, 20, 30, 40, 50
Silver	0, 0.9, 3, 4.5, 6, 7.5
Thallium	0, 4, 10, 20, 30, 40
Tin	0, 4, 20, 40, 80, 100

GFAA / FLAA / CVAA Data Review checklist			Method: 200.9 7000 series AA 245.1 7470/7471		
Instrumentation: PE SIMAA 6000 Varian Spec-20 Varian Spec-10					
Analysis Date:	Data File:		Date Review:		**Analyte:**_____
Cal Std ID:	LIMS # :	Analyst:	Reviewer:		Approved? Yes No

Calibration Parameters -	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV 90-110% 95-105%					
3) ICB ABS of LOD or RL					
6) CRA - (2X CRDL or LOD) 80-120%					
7) CCV1/CCB1- 90-110% / 80-120%					
8) CCV2/CCB2 ABS of LOD or RL					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
12) CCV6/CCB6					

Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	**Comments:**
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					

CT Laboratories
Baraboo Laboratory Division

Title: **Analysis of PAHs by GC/MS SIM**

SOP Number: 8270 SIM PAHs

Written by: _____

Technical Review by: _____

Approved by: _____
Quality Assurance Manager

Approved by: _____
Laboratory Director

SOP Manual Control Number: _____

1.0 SCOPE/APPLICATION

1.1 This method is used in the determination of solvent-extractable semivolatile organic polycyclic aromatic hydrocarbons (PAHs) in many types of solid waste matrices, soils, and groundwater. Target compounds determined by this method are listed in Table 1.0

1.2 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

Table 1.0
Target compounds with Detection Limits and Reporting Limits

PK#	***Compound Name***	***Water MDLs(ug/L)***			***Soil MDLs (ug/Kg)***	
		MDLs	***LOQs***		***MDLs***	***LOQs***
4	Naphthalene	0.006	0.021		1.33	4.34
5	2-Methylnaphthalene	0.005	0.018		1.38	4.60
6	1-Methylnaphthalene	0.006	0.020		1.33	4.43
9	Acenaphthylene	0.004	0.014		1.39	4.64
10	Acenaphthene	0.005	0.016		1.42	4.72
11	Fluorene	0.004	0.014		1.56	5.20
13	Phenanthrene	0.004	0.015		1.58	5.25
14	Anthracene	0.004	0.015		1.50	4.98
15	Fluoranthene	0.005	0.015		1.56	5.21
17	Pyrene	0.004	0.012		1.48	4.93
19	Benzo (a) anthracene	0.004	0.015		1.44	4.81
20	Chrysene	0.004	0.012		1.53	5.11
22	Benzo (b) fluoranthene	0.006	0.021		1.43	4.78
23	Benzo (k) fluoranthene	0.006	0.020		1.48	4.95
24	Benzo (a) pyrene	0.007	0.022		1.37	4.57
25	Indeno (1,2,3-cd) pyrene	0.006	0.021		1.38	4.61
26	Dibenz (a,h) anthracene	0.005	0.018		1.46	4.87
27	Benzo (g,h,i) perylene	0.007	0.022		1.43	4.75

Table 1.0
8270 SIM PAH Compound List

Codes (Tables 1):
S = Surrogates
I = Internal Standards
TM= Target Compounds
CCC= Calibration Check Compounds

PK#	Compound	Retention Time	Relative RT	Primary Ion	Secondary Ion	Code
1	1,4-Dichlorobenzene d_4	3.310	1.00	152		I
2	Naphthalene d_8	4.128	1.00	136		I
3	Nitrobenzene d_5	3.654	0.885	82		S
4	Naphthalene	4.143	1.001	128	127	TM
5	2-Methylhaphthalene	4.613	1.117	142	141	TM
6	1-Methylnaphthalene	4.682	1.134	142	141	TM
7	Acenaphthene d_{10}	5.344	1.00	164		I
8	2-Fluorobiphenyl	4.865	0.910	172		S
9	Acenaphthylene	5.247	0.982	152	151,153	TM
10	Acenaphthene	5.366	1.004	153	152	CCC
11	Fluorene	5.732	1.072	166	165	TM
12	Phenanthrene d_{10}	6.400	1.00	188		I
13	Phenanthrene	6.414	1.002	178	176	TM
14	Anthracene	6.451	1.008	178	176	TM
15	Fluoranthene	7.256	1.134	202	101	CCC
16	Chrysene d_{12}	8.411	1.00	240		I
17	Pyrene	7.423	0.882	202	101	TM
18	Terphenyl d_{14}	7.519	0.894	244		S
19	Benzo(a)anthracene	8.400	0.999	228	226	TM
20	Chrysene	8.438	1.003	228	226	TM
21	Perylene-d_{12}	10.148	1.000	264		I
22	Benzo(b)fluoranthene	9.626	0.948	252	125	TM
23	Benzo(k)fluoranthene	9.633	0.949	252	125	TM
24	Benzo(a)pyrene	10.072	0.993	252	125	CCC
25	Indeno(1,2,3-cd)pyrene	11.407	1.124	276	138	TM
26	Dibenz(a,h)anthracene	11.429	1.126	278	139	TM
27	Benzo(g,h,i)perylene	11.735	1.156	276	138	TM

Table 1.1
Internal Standard For Each Target Compound

1,4-Dichlorobenzene d_4	Naphthalene d_8	Acenaphthene d_{10}	Phenanthrene d_{10}	Chrysene d_{12}	Perylene d_{12}
	Nitrobenzene d_5	2-Fluorobiphenyl	Phenanthrene	Pyrene	Benzo(b)fluoranthene
	Naphthalene	Acenaphthylene	Anthracene	Terphenyl d_{14}	Benzo(k)fluoranthene
	2-Methylnaphthalene	Acenaphthene	Fluoranthene	Benzo(a)anthracene	Benzo(a)pyrene
	1-Methylnaphthalene	Fluorene		Chrysene	Indeno(1,2,3-cd)pyrene
					Dibenz(a,h)anthracene
					Benzo(g,h,i)perylene

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous and soil matrices (reference methods SW846-3510 and 3545), concentration techniques that are suitable for preparing the extract, and the quantitative/qualitative analysis for the determination of target analytes by method SW846-8270.

2.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method applies for aqueous samples extracted by liquid-liquid separatory funnel (SW846-3510). Method applies for soil/sediment and solid waste samples extracted by standard solvent extraction methods utilizing pressurized extraction techniques as heated pressurized fluid extraction (SW846-3545).

2.3 The resultant extract is chemically dried and concentrated in a Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

2.4 Extracts for 8270 analysis may be subjected to Silica Gel (SW846-3630), GPC (SW846-3640A), or Acid Base partition (SW846-3650B) cleanup designed specifically for target analytes. After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with a mass spectrometer detector.

2.5 Identification of target analytes is accomplished by comparing their mass spectra with the spectra of certified commercially-prepared stock standards. Quantitation is accomplished by comparing the response of a major quantitation ion relative to an internal standard using a minimum of a five point calibration curve.

2.6 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

3.0 DEFINITIONS

3.1 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.2 Laboratory Control Spike (LCS): Milli-Q water (for water) or Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike is used to document the ability of an analyst to generate acceptable precision and bias, to verify the analytical system performance, and to document method accuracy for each matrix.

3.3 Laboratory Control Spike Duplicate (LCSD): A duplicate Milli-Q water (for water) or Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike duplicate is used to document the precision and bias of a method. This QC type is only performed when sufficient amount of sample is unavailable for performing a matrix spike and matrix spike duplicate.

3.4 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the precision and bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): Intra-laboratory split samples spiked with identical concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the precision and bias of a method in a given sample matrix.

3.6 Method Reporting Limit (MRL): The method reporting limit is a threshold value below which the laboratory reports a result as a non-detect. The highest value reported for the method reporting limit is dependant upon project-specific action or decision levels. Method reporting limits are adjusted based on the sample matrix and any sample dilution/concentrations when necessary.

3.7 Method Detection Limit (MDL): The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. Depending on project, specific MDLs may be in the form of an MDL check standard or the MDL may be subject to extraction procedures.

3.8 Surrogate (SURR): Organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Its use is to monitor the performance of the extraction, cleanup (as needed), the analytical system, and the effectiveness of the method. The surrogates are: Nitrobenzene-d_5, 2-Fluorobiphenyl, and Terphenyl-d_{14}.

3.9 Initial Calibration (ICAL): An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of an analyte. This relationship may be depicted graphically, and referred to as a "calibration curve". Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte.

3.10 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed.

3.11 Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. A CCV must be analyzed at the beginning of each 12 hour shift.

3.12 DFTPP: Decafluorotriphenylphosphine. This compound is used in tuning the GC/MS. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Benzidine must have a tailing factor less than 2. Pentachlorophenol must have a tailing factor less than 2. Breakdown of DDT

to DDD and DDE must be less than 20%.

3.13 Internal standard (ISTD): Internal Standard involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample extract prior to injection. The ratio of the peak area of the target compound in the sample extract to the peak area of the internal standard in the sample extract is compared to a similar ratio derived for each calibration standard. The internal standards are; 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12} and Perylene-d_{12}.

3.14 Calibration Check Compounds (CCC): Calibration check compounds are a part of the Continuing Calibration Verification standard (CCV). A CCV must be analyzed at the beginning of each 12 hour shift. The CCCs percent difference must be less than or equal to 20%.

3.15 Instrument Blanks (IB): Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank (methylene chloride + Internal Standards) to check for cross-contamination.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method for specific guidance on quality control procedures.

4.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials

4.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes.

4.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample may be necessary.

5.0 SAFETY

5.1 Safety glasses, gloves, and protective clothing: long pants, leather shoes and lab coat or apron should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems. Follow all items in the in-house Chemical Hygiene Plan and Health and Safety Manual.

5.2 The toxicity of chemicals used in this method has not been precisely defined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

6.0 APPARATUS & MATERIALS

Apparatus

6.1 GC-MS system - Hewlett Packard 6890 GC/7683 autosampler/5973 MSD - An analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including syringes, analytical column, mass spectrometer detector, auto sampler, electronic pressure control, vacuum pumps, and HP Chemstation data acquiring system.

6.1.1 Carrier Gas: He at 1.6 mL/min, Constant Flow
Injector Temperature: 250º C
Mode: Pulsed Splitless
Inj. Volume: 0.5 uL
Pressure: 13.2 psi
Pulse Pressure: 30.0 psi
Pulse Time: 0.4 min
Purge Flow: 50.0 ml/min
Purge Time: 0.38 min
Total Flow: 53.9 ml/min
Gas Saver On: 20ml/min at 2.0 min
Oven: Initial – 45º (hold for 0.4 min)
Ramp – 35º/min
Final – 290º
Ramp – 4º/min
Final – 300º
Ramp – 30º/min
Final – 325º (hold for 2.0 min)

6.1.2 MS Interface: 300º
MS Source: 280º
SIM Parameters: See Table 2.0

Table 2.0
Method SIM Parameters

Group	Compounds	Start Time	m/z	dwell
1	1,3	2.20	82,152	50
2	2,4	3.95	82,127,128,136	50
3	5,6,8	4.40	141,142,172	100
4	9,7,12	5.10	151,152,153,164	50
5	11	5.55	165,166	100
6	12,13,14	6.00	176,178,188	50
7	15,17,18	6.80	101,202,244	100
8	19,16,20	7.90	226,228,240	100
9	22,23,24,21	9.00	125,252,264	100
10	25,26,27	10.80	138,139,276,278	50

6.2 GC Column: 30m x 0.25 mm ID, 0.25 um. (J&W DB-5.625 or equivalent).

6.3 Water bath – heated and capable of accepting a Kuderna-Danish apparatus.

6.4 Dionex ASE 200 Accelerated Solvent Extractor.

6.5 Organomation Nitrogen blowdown concentrator.

6.6 Analytical balance capable of accurately weighing to the nearest 0.01 gram (for sample preparation).

6.7 Oven.

Glassware:

6.8 Separatory funnel - 2000 mL glass with Teflon stopcock

6.9 Kuderna-Danish (K-D) apparatus:
- 6.9.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).
- 6.9.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).
- 6.9.3 Snyder column- Three-ball macro (Fisher # K503000-0121).
- 6.9.4 Springs or Teflon clamps to attach concentrator tube to evaporation.

6.10 Graduated cylinder (Class A) - 1000 mL. (Fisher 08-559G).

6.11 Beaker - 250 mL and 600 mL.

6.12 Vials - 2.0 mL and 60 mL amber screw cap vials with Teflon lined caps (Fisher # 03-377-10B).

6.13 Pasteur Pipets (Corning # 7095B-5X).

6.14 Funnels – glass

6.15 Volumetric flask – (Class A) 2, 5, 10, 25, 50, and 100 mL.

6.16 Syringes 10 uL, 25 uL, 100 uL, 500 uL, and 1,000 uL.

Reagents

6.17 Deionized water (Milli-Q processed).

6.18 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212). Purify by heating to 400°C in a shallow glass tray, loosely covered with foil.

6.19 Silica sand- hydrocarbon free. Purify by heating to 400°C in a shallow glass tray, loosely covered with foil.

6.20 Methylene chloride, pesticide grade.

6.21 Acetone, pesticide grade.

6.22 Methanol, pesticide grade.

6.23 Sulfuric Acid (Certified ACS)/Deionzied Water-1:1(v/v).

6.24 Sodium Hydroxide- 10 N (Certified ACS).

6.25 Diatomaceous earth (JT Baker # 1939-01).

6.26 Nitrogen (4.8 rating).

6.27 Helium (4.7 rating).

NOTE: Chemicals are stored at room temperature and in solvent cabinets. The solvents are used in well-ventilated areas. The organic solvents have a shelf life of one year from the date of receipt.

Materials

6.28 Boiling chips, carborundum, approximately 10/40 mesh (methylene chloride rinsed) (G. Frederick Smith Chemical Company, Catalog #198).

6.29 pH indicator paper- pH 0-14 (Whatman Catalog #2613991).

6.30 Dionex ASE Filters (Catalog #049458).

6.31 Filter- Glass Microfibre 12.5 cm GF/A (Whatman # 1827-125).

6.32 Aluminum foil.

6.33 Spatulas- stainless steel.

7.0 STANDARDS AND SPIKES

7.1 Preparation of standards is documented in the GC/MS standards logbook. Each standard is labeled by prep date to allow for tracking. Opened stock standards expire one year or sooner if comparison with quality control check samples indicate a problem. Leftover stock standards are saved in a capped vial in the original box in the freezer. Any subsequent dilutions made from the opened vial expire six months from the original opening. The cracking date of the stock standard vial will be recorded on the label along with the six month expiration date.

7.2 Stock Standards – Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at –10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Opened stock standards expire one year or sooner if comparison with quality control check samples indicate a problem (Not to exceed the manufactures expiration date).The following list of stock standards are commercially prepared standards, which are certified by the manufacturer, such as;

8270 Cal Mix#6 : Restek #31622 at 2000 ug/ml
8270 Surrogate Mix BN: Restek # 31062 at 5000 ug/ml
DFTPP: Restek Part # 31615 at 1000 ug/ml

7.3 Intermediate Stock Standards: These standards are diluted stock standards so that the concentration levels are manageable for the preparation of working standards. The 8270

intermediate standard is prepared at an optimum level for the preparation of the working stock standard. Each 8270 target compound is at a concentration of 20.0 ug/ml with surrogate concentration at 20.0 ug/ml in methylene chloride.

Table 3.0
Intermediate Stock Standard

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 Cal Mix#6	2000	0.100	10.0	20.0
8270 BN SURR	5000	0.040	10.0	20.0

7.4 Calibration standards: An initial calibration of the listed analytes in Table 1.0 is performed using a minimum of 5 points. The following concentrations correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. Standards are made by taking aliquots of the intermediate standard and diluting to volume in methylene chloride. The following levels are repeated across all compounds.

Level 1	20 ng/ml
Level 2	50 ng/ml
Level 3	100 ng/ml
Level 4	500 ng/ml
Level 5	1000 ng/ml
Level 6	1500 ng/ml
Level 7	2000 ng/ml

Table 3.1
Initial Calibration Points for the 8270 Linearity

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ng/ml)
1	20.0	10	10.0	20
2	20.0	25	10.0	50
3	20.0	50	10.0	100
4	20.0	250	10.0	500
5	20.0	500	10.0	1000
6	20.0	750	10.0	1500
7	20.0	1000	10.0	2000

7.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target compounds at 500 ng/ml each time the initial calibration is performed. Standards are made by taking aliquots of the ICV intermediate standard and diluting to volume in methylene chloride. The ICV stock standard is prepared in the same manner as the primary intermediate stock standard. The ICV working standard will be prepared

at the time of initial calibration and should have a shelf live of one week.

Table 3.2
ICV Working 8270 Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ng/ml)
ICV	20.0	250	10.0	500

7.6 Calibration Verification Standard (CCV): A working standard solution for 8270 at a concentration of 500 ng/ml. It is used to check the validity of a calibration curve on a daily basis. Standard is made by taking an aliquot of the intermediate standard and diluting it to volume in methylene chloride. The CCV should be prepared weekly and stored at 4ºC.

Table 3.3
CCV Working 8270 Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ng/ml)
8270 SIM PAH	20.0	250	10.0	500

7.7 Surrogate standard: A commercially prepared certified solution of Nitrobenzene-d_5, 2-Fluorobiphenyl, and Terphenyl-d_{14} is diluted in methanol to produce a working surrogate solution of 2 ug/mL. 1.0 mL is added to each sample. The surrogate concentration is normalized to 100% from the spiking solution in the CCV level (point 4) of the initial calibration. This will provide percent recoveries that transfer directly to LIMS.

Table 3.4
Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
8270 BN SURR	5000	20.0	100.0	1.0

7.8 Spiking standards (matrix and control samples): Prepare a spiking solution in methanol that contains target compounds for water and sediment / soil samples. 1.0 ml is added to quality control and matrix spike samples.

Table 3.5

Compound Spiking Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 SIM PAH	2000	0.050	100	1.0

7.9 DFTPP: Decafluorotriphenylphosphine. This compound is used in tuning the GC/MS. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness.

Table 3.6
DFTPP Standard Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
DFTPP	1000	0.250	10.0	25.0

NOTE: All standards are stored at -10ºC. Opened stock standards expire one year or sooner if comparison with quality control check samples indicated a problem. Intermediate standards expire six months from the date made. An intermediate stock standard or working standard shall not exceed expiration date criteria. All subsequent standards made from the intermediate stock standards expire on the same date as the working stock standard. If more than one standard is added to a solution the expiration date will be the same as the stock standard with the earliest expiration date

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples should be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. The soil samples must be extracted within 14 days and water samples must be extracted in 7 days, from the date of collection.

8.2 Sample extracts should be stored under refrigeration in the dark and analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

9.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4 laboratory control spikes for each matrix and sample type. After analysis,

the analyst calculates the average recovery (x) in µg/L and the relative standard deviation (RSD) of the recovery 8270 target compounds. In addition to each set of IDCs, a blind laboratory spike will be performed. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000, sec. 8.4.9).

9.3 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SURR, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Client and/or Project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 4) also lists recovery limits specific to the method/project/program.

10.0 CALIBRATION

10.1 Calibration

10.1.1 The initial calibration for SW-846 chromatographic methods involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument.

10.1.2 For each compound and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methylene chloride.

10.1.3 The lowest concentration calibration standard, that is analyzed during an initial calibration curve, establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

10.1.4 Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the response of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area or height of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

10.1.4.1 Internal standards are recommended in SW846-8270. These internal standards are: 1,4-Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12}. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

10.1.4.2 In preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the

concentrations of the target analytes will vary. 10uL of a solution containing the internal standards at a concentration of 100 ug/mL is added to each 1 mL of standard or sample extract. This results in an internal standard concentration of 1.0 ug/mL on the instrument. The mass of each internal standard added to each sample extract immediately prior to injection into the instrument must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs (e.g., 10 ul of solution added to a 1 ml final extract results in only a negligible 0.1% change in the final extract volume which can be ignored in the calculations).

10.1.4.3 An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response (area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound.

10.1.5 For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows;

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Concentration of the analyte or surrogate in ug/mL.

C_{is} = Concentration of the internal standard in ug/mL.

10.1.6 Linear calibration using the average response factor. Response factors are a measure of the slope of the calibration relationship and assume that the curve passes through the origin. Under ideal conditions, the factors will not vary with the concentration of the standard that is injected into the instrument. In practice, some variation is to be expected. However, when the variation, measured as the relative standard deviation (RSD), is less than or equal to 15%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. To evaluate the linearity of the initial calibration, calculate the RF, the standard deviation, and the relative standard deviation.

$$\text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (RF_I + \overline{RF})^2}{n-1}\right)}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

10.1.7 The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C sec. 11.5). When ARFs are not acceptable, results are sometimes calculated using linear (1st order) regression curves and/or quadratic (2nd order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5).

10.1.8 Linear Calibration: If the RSD of the calibration factor is greater than 15% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst may employ a regression equation that does not pass through the origin. This approach may also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

10.1.9 The use of origin (0,0) as a calibration point is not allowed. However, most data systems and many commercial software packages will allow the analyst to "force" the regression through zero. This is not the same as including the origin as a fictitious point in the calibration. It may be appropriate to force the regression through zero for some calibrations (SW-846 Method 8000C sec. 11.5.2.1). The use of linear regression may not be used as a rationale for reporting results below the calibration range.

10.1.10 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.1.11 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Linear regression employs five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards. The coefficient of determination (COD) is calculated as follows:

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p} \right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

y_{obs} = Observed response (area) for each concentration of the calibration curve.

$\bar{y}$ = Mean observed response from the initial calibration.

Y_I = Calculated response at each concentration from the initial calibrations.

n = Total number of calibration points (6 points for quadratic equation).

p = Number of adjustable parameters in the polynomial.

10.1.12 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of determination will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99. Weighting in a calibration model may significantly improve the ability of the least squares regression to fit the data calibrations (SW-846 Method 8000C sec. 11.5.3).

10.2 Calibration Criteria

10.2.1 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so a 25 ng injection of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 3. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

Table 3.0
DFTPP Mass Spectrum

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
69	<100% of mass 198
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1.0% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

10.2.2 To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP.

10.2.3 The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal responses and peak tailing should be evaluated. Benzidine should have a tailing factor less than 2, and Pentachlorophenol should have a tailing factor less than 2.

10.2.4 Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels and are prepared from the working standard dilutions of intermediate stock standards. One of the concentration levels should be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples and should contain each analyte for detection by this method. If the measured relative standard deviation (RSD) is less than or equal to 15%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. Linear Calibration: If the RSD of the calibration factor is greater than 15% over the calibration range, then linearity though the origin cannot be assumed. In this case, the analyst may employ a regression equation that does not pass through the origin. This approach may also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms

for a linear equation. In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range.

10.2.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed. The percent difference of the analytes must be less than or equal to 20%.

10.2.6 Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The percent difference of the analytes must be less than or equal to 20%.

10.2.7 The relative retention time (RRT) of each compound in each calibration standard should agree within 0.06 RRT units.

10.2.8 Sporadic Marginal Failures: The number of target analytes reported for the method will dictate the number of allowable QC failures as given below. The sporadic marginal failure allowance entails the application of an expanded acceptance criterion. If these QC limits are not met, a new initial calibration must be performed.

Table 3.1
Sporadic Marginal Failures

N[1]	X[2]
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

11.0 PREPARATION PROCEDURES

11.1 Water Extraction (Method SW-846, 3510)

11.1.1 Pre-rinse all glassware to be used in the extraction with methylene chloride (Pesticide Grade).

11.1.2 Mark the meniscus on the bottle for later determination of sample volume (see sec 11.1.11). From the glass sample collection bottle, quantitatively transfer sample into a 2 liter separatory funnel.

11.1.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding one liter of Milli-Q water to 2 liter separatory funnel.

11.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and from the glass sample collection bottle, quantitatively transfer half of the sample into a 2 liter separatory funnel and label MS, and transfer the other half of the sample into another 2 liter separatory funnel and label MSD. If there is no sample to perform a matrix spike/matrix spike duplicate a laboratory control spike duplicate must be performed.

11.1.5 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 1.0 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the 8270 SIM Exp. spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.1.6 Add 60 mLs of methylene chloride to the sample's separatory funnel. Extract the sample shaking vigorously for two minutes, venting frequently.

11.1.7 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Decant the lower layer into a 500 ml beaker. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.1.8 Repeat the extraction two more times using fresh 60 mL portions of methylene chloride.

11.1.9 Determine the sample volume by filling the sample bottle to the mark (11.1.2) with water and transferring it to a "Class A" 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet.

11.1.10 Refer to section 11.3 for sample concentration.

11.2 Soil Extraction (Method SW-846, 3545)

11.2.1 Preparing the extraction cell for use: Pre-rinse extraction cell (tube and caps) with methylene chloride. Attach the screw fit tube cap of the soil extractor to the end of the tube without the serial number engraved on the side. Using the filter rod, push 1 filter through the open end of the tube until they reside flush on the bottom of the screwed end.

11.2.2 Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1mm sieve or can be extruded through a 1mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make sample more amenable to grinding.

11.2.4 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make sample easier to mix.

11.2.5 (Refer to SOP FO-10 for subsampling guidance). Weigh approximately 10 g of sample to the nearest 0.1 g into a 250-mL beaker and record the final weight. Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples should be a free flowing powder. If sample is not free flowing add more diatomaceous earth until the sample has a dry texture. This powder is so mixed that it will allow the sample to pass through a 2 mm sieve.

11.2.6 Transfer the ground sample to an extraction cell of appropriate size for the aliquot.

11.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10g of sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

11.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh three individual 10 grams aliquots of sample. Add drying agent. Transfer sample to extraction cell. If there is no sample available to perform a matrix spike/matrix spike duplicated a laboratory control spike duplicate must be performed.

11.2.9 Fill the void in each of the extraction cells with clean sand.

11.2.10 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 1.0 mL of the 8270 surrogate standard solution by using a 1.0 ml syringe. In addition, add 1.0 mL 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.2.11 Attach the other cap to the other end of the extractor cell, making sure the tube is sitting flush on a hard surface so that no particulates get caught in the threads of the tube cap.

11.2.12 Place the extractor tube on the Dionex on a selected position with the serial number engraved end up on the top wheel. Place an empty 60 mL VOA collection vial, labeled with the sample ID, on the matching position on the bottom wheel. Schedule the Dionex ASE 200 and begin the cycle.

11.2.13 The Dionex ASE 200 extraction cycle:

Oven temperature: 100 º C Pressure: 1500 psi
Prepurge time: 0 minutes
Static time: 6 minutes
Heat: 5 minutes
Flush volume: 65%
Nitrogen purge: 60 sec. At 150 psi
Solvent A: 100%
Method rinse: ON
Static Cycles: 1
Extraction Fluid: (70:30) methylene chloride : acetone

11.2.14 Refer to section 11.3 for sample concentration.

11.3 Sample Concentration

11.3.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds of the depth with Na_2SO_4. Rinse filter paper, Na_2SO_4, funnel, K-D apparatus, and concentrator tube with methylene chloride.

11.3.2 Quantitatively pour the extract through the filter and funnel seated on a concentrating, 500 ml Kuderna-Danish (K-D) for water samples or a 250mL Kuderna-Danish (K-D) for soil samples, apparatus complete with concentrator tube. Rinse the beaker three times with methylene chloride. Add these rinses through the filter and funnel into the K-D apparatus. Add a boiling chip to the K-D flask prior to placing it on the heated water bath. Wet a three ball Snyder column with approximately 2 mL of methylene chloride. Attach the Snyder column.

11.3.3 Place the K-D in the heated water bath so the concentrator tube is immersed in the water and the lower rounded surface of the K-D is bathed in steam. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood (set the knob of the temperature control to 5~6). It is critical that the analyst watch the extract as it distills. THE EXTRACT SHOULD NOT GO TO DRYNESS.

11.3.4 When the extract volume reaches approximately 5-7 mL, remove the K-D from the bath. Slightly tilt the apparatus and rotate to aid in solvent drainage from the Snyder column. Allow it to cool completely.

11.3.5 Remove the Snyder column, rinse the ground glass joints with a small amount of methylene chloride and then remove K-D flask. Turn on the heating unit for the Organomation. The water bath should be about 35ºC. Place sample concentrator tube into the nitrogen blow down apparatus. Allow a gentle stream of nitrogen to interact with the extract. There should be no splashing or excessive movement upon the surface of the extract. Allow the extract to evaporate down to 0.8 ml. Remove concentrator tube from water bath and by using a Pasteur pipet, bring sample extract up to 1.0 ml volume with methylene chloride.

11.3.6 Transfer the 1 mL of the extract to a labeled amber screw-cap injection vial. Record the final extract on the injection extraction bench sheet.

11.3.7 The sample extract is now ready for analysis. If samples are not analyzed immediately store the sample extract in a refrigerator.

11.3.8 Sample may require cleanup prior to analysis. Refer to attachments I, II, or III for sample cleanup options.

12.0 SAMPLE ANALYSIS

12.1 Sample Sequence

12.1.1 It is highly recommended that sample extracts be screened on a GC/FID to protect the GC/MS system from unexpectedly high concentrations of organic compounds.

12.1.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 ul of the internal standard solution to the 1 ml concentration sample extract obtained from sample preparation.

12.1.3 Before initial calibration or sample analysis a priming standard may be injected at a level 2x the highest linearity point.

12.1.4 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so a 25 ng injection of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 3. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal responses and peak tailing is evaluated. Benzidine must have a tailing factor less than 2 and Pentachlorophenol must have a tailing factor less than 2. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

12.1.5 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing target analytes, prior to conducting any sample analysis. A CCV must be injected at the begining of each twelve hour shift. The variance of any given target analyte should not be more than +/- 20% difference. The percent difference of the CCCs must be less than or equal to 20%.

12.1.6 The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the CCV level from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCVs and associated samples while the system was malfunctioning is necessary

12.1.7 Samples can be directly injected after the successful analyses of the initial calibration curve, ICV, DFTPP, and CCV. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control

spike must be analyzed with every analytical batch. Recoveries should be within laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections. Some sample extracts may require clean-up procedures. Refer to attachments I and II.

12.1.8 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS, the sample extract must be diluted and reanalyzed. Additional internal standards must be added to the diluted extract to maintain the same concentration as in the calibration standards.

12.1.9 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method (SW-846-8270). The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

12.1.9.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

12.1.9.2 The relative retention time (RRT) of each compound in each calibration standard should agree within 0.06 RRT units.

12.1.9.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.1.9.4 Structural isomers that produce very similar spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomeric peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs that may be separable by the GC should be identified, quantitated and reported as the sum of both compounds by the GC.

12.2 Sample Calculations

12.2.1 Re-arranging the equation from sec. 10.1.5 to calculate the "as-analyzed" value yields:

$$C_s = \frac{A_s \times C_{is}}{A_{is} \times RF}$$

RF = Average Response Factor

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Concentration of the analyte or surrogate in ug/mL.

C_{is} = Concentration of the internal standard in ug/mL.

12.2.2 Once the target components of the extract have been identified and quantitated, the "as-analyzed" value is converted to the "as-received" concentration as follows:

Water Matrix:

$$\frac{(ug/mL\ injected)\,x\,(mL\ extract\ \ final\ volume)x(dilutionfactor)}{(volume\ of\ \ sample\ extracted, in\ L)} = \mu g/L$$

Soil Matrix:

$$\frac{(ug/mL\ injected)\,x\,(mL\ extract\ \ final\ volume)x(dilutionfactor)}{(weight\ of\ \ sample\ extracted, in\ g)} = ug/g$$

13.0 Data Assessment & Acceptance Criteria for QC Measures.

13.1 Data Assessment & Acceptance Criteria for QC Measures.

13.1.1 If the initial analysis of a sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be re-analyzed at a dilution.

13.1.2 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method SW-846-8270.

13.1.3 The relative retention time (RRT) of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

13.2 Reporting Quantitative Analysis

13.2.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times and mass ions are compared to the sample retention times, the sample data can be reported.

13.2.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 5.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs the checklist. Senior analysts have the authority to validate their own data. Finally, the validated results are made available to the client services personnel.

13.2.3 An electronic copy of the data is then filed or archived. The package includes the checklist, the sequence run log, the LIMS run log, verification of calibration data and chromatograms/quant reports. All the data is to be initialed and dated by the analyst. Each data file header should contain the Prep ID #, date prepped,

Analytical ID #, and date of sequence.

13.3 Corrective Measures for Out-of-Control Data

13.3.1 When data is out of control, corrective actions are required. Certain situations will cause data deficiencies, for example: if the non-conformities involve failing QC within the analytical sequence batch, the reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination, and the sample may need diluting or undergo sample clean-up procedures to reduce matrix effect. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or non-conformities (Table 4.)

13.4 Contingencies for Handling Out-of-Control or Unacceptable Data

13.4.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their effect on data.

13.4.2 To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises re-analysis or re-extraction is an option.

13.4.3 If a sample is extracted passed holding times, the analyst must notify client services immediately. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur. If the client still request sample analysis, the sample data is qualified for holding time violations.

13.4.4 If the non-conformities involve failing QC within the analytical sequence batch, the analyst must reanalyze the extract. This will ensure that the instrument was functioning properly. If the results are passing, the non-conformities were associated with the instrument. If data is still outside QC criteria, re-extraction of the all samples associated with the QC samples must be performed (provided that there is sample available). If no sample is available for re-extraction, the samples are qualified accordingly.

13.4.5 If the out of control data is the result of instrument malfunctions, the analyst must evaluated and repair instrument prior to reanalysis of the affected data. After the instrument maintenance and repair of the downed instrument which caused data to be out of control must demonstrate corrective measures by analyzing a Continuing Calibration Verification (CCV) before continuing sample analysis. Any maintenance and repair must be noted in the instrument logbook.

13.4.6 If sample matrix affect or contamination is the reason for poor data, the analyst will use professional judgement for the reanalysis of the extract. The extract can be diluted and reanalyzed. The sample can be re-extracted confirming matrix

interference. The analyst can report and qualify the sample data. The analyst must supply explanation for the qualified data in the narrative or client notes.

13.4.7 If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

13.4.8 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

13.4.8.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

13.4.8.2 In all instances, if results are reported from data that is out of control or unacceptable, that data is qualified accordingly. Once the client has been notified and he or she instructs us to report the data, then flag the data indicating what type of nonconformity has occurred.

13.4.8.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data.

13.4.8.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

14.0 WASTE MANAGEMENT

14.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

ATTACHMENT I
ACID-BASE PARTITION CLEANUP
METHOD 3650B

1.0 SCOPE AND APPLICATION

1.1 Method 3650 is a liquid/liquid partitioning cleanup method to separate acid analytes from base/neutral analytes using pH adjustments. It may be used for cleanup of petroleum waste prior to analysis or futher cleanup.

Compounds by Fraction

Compound	Fraction
Benzo (a) anthracene	Base-Neutral
Benzo (a) pyrene	Base-Neutral
Benzo (b) fluoranthene	Base-Neutral
Chlordane	Base-Neutral
2-Chlorophenol	Acid
Chrysene	Base-Neutral
Cresol(s)	Acid
Dichlorobenzene(s)	Base-Neutral
2,4-Dimethylphenol	Acid
Dinitrobenzene	Base-Neutral
4,6-Dinitro-2-Methylphenol	Acid
2,4-Dinitrotoluene	Base-Neutral
Hexachlorobenzene	Base-Neutral
Hexachlorocyclopentadiene	Base-Neutral
Naphthalene	Base-Neutral
Nitrobenzene	Base-Neutral
4-Nitrophenol	Acid
Pentachlorophenol	Acid
Phenol	Acid
Pyridine	Base-Neutral
Tetrachlorobenzene(s)	Base-Neutral
Tetrochlorophenols(s)	Acid
Trichlorophenol(s)	Acid

1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The solvent extract from a prior extraction method is shaken with water that is strongly basic. The acid analytes partition into the aqueous layer, whereas, the basic and neutral compounds stay in the organic solvent. The base/neutral fraction is concentrated and is then ready for further cleanup, if necessary, or analysis. The aqueous layer is acidified and extracted with an organic solvent. This extact is concentrated and is then ready for analysis of the acid analytes.

3.0 INTERFERENCES

3.1 More extensive procedures than those outlined in this method may be necessary for reagent purification.

3.2 A method blank must be run for the compounds of interest prior to use of the method. The interferences must be below the method detection limit before this method is applied to actual samples.

4.0 APPARATUS AND MATERIALS

4.1 Drying column –20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom, or equivalent.

4.2 Autosampler vials.

4.3 Heated water bath.

4.4 Boiling Chips.

4.5 pH paper (range 0-14).

4.6 Separatory Funnel.

4.7 Erlenmeyer Flask, 125 ml.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water.

5.3 Sodium Hydroxide, (10N)

5.4 Sulfuric Acid, (1:1 v/v in water)

5.5 Sodium Sulfate (Purified by heating to 400º).

5.6 Methylene Chloride, pesticide grade.
5.7 Acetone, pesticide grade.

5.8 Methanol, pesticide grade.

5.9 Diethyl Ether, pesticide grade.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to Method 8270, Section 8.0 for sample handling.

7.0 PROCEDURE

7.1 Place 10 ml of solvent extract from a prior extraction procedure into a 125 ml separatory funnel.

7.2 Add 20 ml of methylene chloride to the separatory funnel.

7.3 Slowly add 20 ml of prechilled organic-free reagent water which has been previously adjusted to a pH of 12-13 with 10N sodium hydroxide.

7.4 Seal and shake the separatory funnel for at least 2 minutes with periodic venting to release excess pressure.

7.5 Allow the organic layer to separate from the aqueous phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration, centrifugation, or other physical methods.

7.6 Separate the aqueous phase and transfer it to a 125 ml Erlenmeyer flask. Repeat the extraction two more times using 20 ml aliquots of dilute sodium hydroxide. Combine the aqueous extracts.

7.7 Water soluble organic acids and phenols will be primarily in the aqueous phase. Base/Neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase, discard the methylene chloride and proceed to Section 7.8. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 7.10.

7.8 Externally cool the 125 ml Erlenmeyer flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Quantitatively transfer the cool aqueous phase to a clean 125 ml separatory funnel. Add 20 ml of methylene chloride to the separatory funnel and shake for at least 2 minutes. Allow the methylene chloride to separate from the aqueous phase and collect the methylene chloride in an Erlenmeyer flask.

7.9 Add 20 ml of methylene chloride to the separatory funnel and extract at pH 1-2 a second time. Perform a third extraction in the same manner combining the extracts in the Erlenmeyer flask.

7.10 Prepare the extracts for sample concentration. Refer to method 8270, Section 9.3 for sample concentration techniques.

8.0 QUALITY CONTROL

8.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples.

8.2 For samples that are cleaned using this method, the associated quality control samples must be processed through this cleanup method.

ATTACHMENT II
GEL-PERMEATION CLEANUP
METHOD 3640A

1.0 SCOPE AND APPLICATION

1.1 Gel-Permeation Chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinylbenzene-styrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

1.2 General cleanup application – GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes; therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

1.3 Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue will ultimately reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem. GPC, operating on the principal of size exclusion, will not usually remove interference peaks that appear in the chromatogram since the molecular size of these compounds is relative similar to the target analytes. Separation cleanup techniques, based on other molecular characteristics (i.e. polarity), must be used to eliminate this type of interference.

1.4 This method is restricted for use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method is used to cleanup extracted samples with unwanted light and/or heavy compounds that interfere with final analysis of methods; 8081, 8082, 8270 and 8310.

2.2 Samples are extracted then concentrated and prepared for GPC cleanup.

2.3 Determinations for which samples that are candidates for GPC cleanup are based on client specific, site specific and project specific requirements, specific

characteristics of initial extract (e.g. color or odor) or interference determined by initial extract analysis.

2.4 The column is calibrated and then loaded with the sample extract to be cleaned up. Elution is effected with a suitable solvent(s) and collection times are adjusted based on desired analysis. The collected fractions are then concentrated per guidelines for methods; 8081, 8082, 8270 and 8310.

3.0 INTERFERENCES

3.1 A reagent blank is to be analyzed for the compound of interest prior to the use of this method. The level of interferences must be below the estimated quantitation limits (EQL's) of the analytes of interest before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method will be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Gel-permeation chromatography system - Gilson GV-271 ASPEC, Gilson, INC or equivalent. All systems, whether automated or manual must meet the calibration requirements.

4.2 Chromatographic column -350mm x 21.20 mm 0 micron, Phenomenex p/n 00W-3035-PO or equivalent.

4.3 Guard column – (optional) 60mm x 21.20 mm 0 micron, Phenomenex p/n 03R-3035-PO or equivalent.

4.4 Ultraviolet detector –fixed wavelength (254 nm) with a semi-prep flow-through cell, Gilson 112 UV/VIS detector, Gilson, INC..

4.5 Strip chart recorder, recording integrator or laboratory data system, (Trilution and LIMS).

4.6 Syringe – 10ml with Luerlok fitting.

4.7 Syringe filter assembly, disposable – Puradisc 25TF sample filter assembly Whatman #6784-2510, 25mm and 1.0 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

4.8 Analytical balance – 0.00 g.

4.9 Volumetric flasks, Class A 5ml to 100ml

4.10 Graduated cylinders

4.11 Disposable glass culture tubes 13mm x 100mm, (Kimble 73500-13100).

4.12 Disposable glass collection tubes- (Kimax 51 25mm x 200mm, Kimble 45060-25200).

4.13 Pasteur pipets- (Corning #7095B-5X)

Reagents

4.14 Methylene chloride, Pesticide grade.

4.15 Cyclohexane, Pesticide grade.

4.16 N-Butyl chloride, Pesticide grade.

5.0 CALIBRATION

5.1 Preparation of standards is documented in the GPC standard logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards once opened expire within six months or sooner if routine QC indicates a problem and not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire in six months and not to exceed the opened date of the stock standard and not to exceed manufactures expiration date.

5.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Stock standards once opened expire in six months and not to exceed the manufactures expiration date.

5.3 The following is the stock standard that is commercially prepared standard, which is certified by the manufacturer;

GPC Calibration Mix: Restek Part # 32019. (1ml ampul)
GPC Calibration Mix: Restek Part # 32023. (5ml ampul)

5.4 GPC Calibration Solution is a certified prepared standard in methylene chloride containing the following analytes (in elution order):

Compound	mg/mL
Corn oil	250

Bis (2-ethylhexyl) phthalate	10
Methoxychlor	2.0
Perylene	0.2
Sulfur	0.8

Table 1.0 GPC Calibration Standard

Standard	Component Name	Conc. (mg/ml)	STD Volume (ml)	Final Volume (ml)	Final Conc. (ug/ml)
GPC STD	Corn Oil	250	5.0	50.0	25000.0
	Bis(2-ethylhexyl)pht.	10	5.0	50.0	1000.0
	Methoxychlor	2.0	5.0	50.0	200.0
	Perylene	0.2	5.0	50.0	20.0
	Sulfur	0.8	5.0	50.0	80.0

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND EXTRACTION PROCEDURES

6.1 Follow guidelines listed in section 8.0 in SOP 8270 Rev. 8 for sample collection, preservation and handling.

7.0 PROCEDURE

7.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 72°F.

7.2 GPC Setup

Column Preparation:

7.2.1 Using manual control options in Trilution set the system to equilibrate for at least 30 minutes prior to starting a run or priming.

7.2.2 If system has not been used on a regular basis, prime all lines and pumps either manually or using the automated system following guidelines listed in the Trilution help manual.

7.3 Calibration of the GPC Column

7.3.1 Place approximately 6 to 7 mls of the calibration solution in a disposable culture tube. Place tube in position 1 on the sample tray. Set Trilution to run a calibration method with an inject volume of 5.50ml.

7.3.2 Following are criteria for evaluation of the UV chromatogram for column condition.

7.3.3 Peaks must be observed and will be symmetrical for all compounds in the calibration solution.

7.3.4 Corn oil and phthalate peaks must exhibit >85% resolution.

7.3.5 Phthalate and methoxychlor peaks must exhibit >85% resolution.

7.3.6 Methoxychlor and perylene peaks must exhibit >85% resolution.

7.3.7 Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

7.3.8 Nitroaromatic compounds are particularly prone to adsorption. For example 4-nitrophenol recoveries can be low due to a portion of the analyte being discarded after the end of collection time. Columns are to be tested with the semivolatiles matrix spiking solution. GPC elution will continue until after perylene has eluted or long enough to recover at least 85% of the analytes, whichever time is longer.

7.3.9 Calibration for Semivolatiles – Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Collection can be stopped before sulfur elutes. Each laboratory is required to establish its specific time sequences.

7.3.10 Calibration for Organochlorine Pesticides/PCBs – Determining the elution times for the phthalate, methoxychlor, perylene and sulfur. Choose a dump time which removes >85% of the phthalate, but collects >95% of the methoxychlor. Stop collection after the elution of perylene, but before sulfur elutes.

7.3.11 Calibration for Polynuclear Aromatic Hydrocarbons – Determine the elution times for corn oil, phthalate and perylene. Start elution collections just before the elution of bis (2-ethylhexyl) phthalate and end collection after the elution of perylene.

7.3.12 Verify the flow rate by collection column eluate for 10 minutes in a graduated cylinder and measure the volume which will between 45-55ml (4.5-5.5 ml/min). If flow rate is outside of this range, corrective action must be taken, as described above. Once flow rate is within the ranges of 4.5-5.5 ml/min, record the column pressure (6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature

conditions can affect analyte retention times and must be monitored. If flow rate and/or column pressure do not fall within the above ranges the column or pre-column are suspected. A UV trace that does not meet the criteria in section 7.2.2.2 would also indicate that the column or pre-column are responsible the poor performance.

7.3.13 Reinject the calibration solution after appropriate collect and dump cycles have been set and the solvent flow and column pressure have been established.

7.3.14 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed can be used to indicate problems with the system during sample processing.

7.3.15 The retention times for bis (2-ethylhexyl) phthalate and perylene must not vary more than ± 5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by:

7.3.15.1 Poor laboratory temperature control or system leaks.

7.3.15.2 An unstabilized column that requires pumping methylene chloride through it for several more hours.

7.3.15.3 Excessive laboratory temperatures, causing outgassing of the methylene chloride.

7.3.16 Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by whatever detectors will be used for the analysis of future samples. Exchange the solvent if necessary. If the blank exceeds the estimated quantitation limit of the analytes, pump additional methylene chloride through the system for 1 to 2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

7.3.17 A hard copy calibration report will follow the samples in the sample prep folder.

8.0 EXTRACT PREPARATION

8.1 Adjust the extract volume to 5 ml. The solvent extract must be primarily methylene chloride. All other solvents, e.g. 1:1 methylene chloride/acetone, must be concentrated to 1 ml (or as low as possible if a precipitate forms) and diluted to 5 ml with methylene chloride. Thoroughly mix the extract before proceeding.

8.2 Filter the extract through a 1 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 ml syringe. Draw the sample extract

through the filter assembly and into the 10 ml syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container, e.g. a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into a syringe without the filter assembly, attach the filter assembly and force the extract through the filter and into the glass container. The latter is the preferred technique for viscous extracts or extracts with a lot of solids. Particulate larger than 5 microns can scratch the valve, which can result in a system leak and cross-contamination of sample extracts in the sample loops.

NOTE**:** Viscosity of a sample extract can not exceed the viscosity of 1:1 water/glycerol. Dilute samples that exceed the viscosity.

9.0 SCREENING THE EXTRACT

9.1 Screen the extract to determine the weight of dissolved residue by evaporating a 100 µL aliquot to dryness and weighing the residue. The weight of dissolved residue loaded on the GPC column cannot exceed 0.500 g. Residues exceeding 0.500 g will very likely result in incomplete extract cleanup and contamination of the GPC switching valve (which results in cross-contamination of sample extracts).

9.1.1 Transfer 100µL of the filtered extract from section 8.2 to a tared aluminum weighing dish.

9.1.2 A suggested evaporation technique is to use a heat lamp. Set up a 250 watt heat lamp in a hood so that it is 8 ± 0.5cm from a surface covered with a clean sheet of aluminum foil. Surface temperature will be between 80-100°C (check temperature by placing a thermometer on the foil and under the lamp). Place the weighing dish under the lamp using tongs. Allow it to stay under the lamp for 1 minute. Transfer the weighing dish to an analytical balance or a micro balance and weigh to the nearest 0.1mg. If the residue weight is less than 10mg/100µL, then further weighings are not necessary. If the residue weight is greater than 10mg/100µL then determine if constant weight has been achieved by placing the weighing dish and residue back under the heat lamp for 2 or more additional 0.5 minute intervals. Reweigh after each interval. Constant weight is achieved when three weights agree within ±10%.

9.1.3 Repeat the above residue analysis on a blank and a spike. Add 100µL of the same methylene chloride used for the sample extraction to a weighing dish and determine residue as above. Add 100µL of a corn oil spike (5g/100ml) to another weighing dish and repeat the residue determination.

9.1.4 A residue weight of 10mg/100µL of extract represents 500mg in 5ml of extract. Any sample extracts that exceed the 10mg/100µL residue weight must be diluted so that the 5ml loaded on the GPC column does not

exceed 0.500g. Following is a calculation that will be used to determine what dilution is necessary if the residue exceeds 10mg.

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{10\text{mg maximum}}{\text{X mg of residue}}$$

Example

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{10\text{mg maximum}}{15 \text{ mg of residue}}$$

Y ml taken for dilution = 3.3 ml

Therefore, taking 3.3 ml of sample extract from 7.4.2 and diluting it to 5ml with methylene chloride will result in 5 ml of diluted extract loaded on the GPC column that contains 0.500 g of residue.

NOTE**:** This dilution factor must be included in the final calculation of analyte concentrations.

10.0 GPC CLEANUP

10.1 Calibrate the GPC at least once per week following the procedure outlined in section 7.3. Ensure that UV trace requirements, flow rate and column pressure criteria are acceptable. Also, retention time shift must be <5% when compared to retention times in the last calibration UV trace.

10.2 If these criteria are not met, follow appropriate maintenance methods to try to regain resolution.

10.3 Load sample vials with 5 ml of filtered extract from section 7.4.2. The Method Blank (MB) and Laboratory Control Standard (LCS) must be run through the GPC process from each prep batch that the samples originated from.

NOTE**:** The number of samples from each prep batch that are put through GPC will vary by client, location and project specifications

10.4 Set the GPC up to run cleanup of the samples, verifying that collection and dump times correlate with the calibration times for each analysis specified in section 7.3. Multiple methods can be used on each GPC run. Follow software specific guidelines for setting up a GPC run.

NOTE**:** It will be necessary to run multiple rinse methods between samples to ensure no cross-contamination between particularly dirty samples

10.5 Monitor sample volumes collected. Changes in sample volumes collected will indicate one or more of the following problems:

10.5.1 Change in solvent flow rate caused by channeling in column or changes in column pressure.

10.5.2 Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

10.5.3 Leaks in the system or significant variances in room temperature.

10.6 Concentrate the extract by following the methods listed in the sections for Sample Concentration for methods; 8081, 8082, 8270 and 8310.

11.0 QUALITY CONTROL

11.1 The analyst will demonstrate that the compound(s) of interest are being quantitatively recovered before applying this method to actual samples.

11.2 For sample extracts that are cleaned up using this method, the associated quality control samples must also be processed through this clean up method.

11.3 Some brands of methylene chloride can contain unacceptably high levels of acid (HCl). Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer.

11.4 If the pH of the water layer is ≤5, filter the entire supply of solvent through a 2 in x 15 in glass column containing activated basic alumina. This column will be sufficient for processing approximately 20-30 liters of solvent. Alternatively, find a different supply of methylene chloride.

ATTACHMENT III
SILICA GEL CLEANUP
METHOD 3630C

1.0 SCOPE AND APPLICATION

1.1 Silica gel is a regenerative adsorbent of silica with weakly acidic properties. Silica gel can be used in column in column chromatography for the separation of analytes from interfering compounds of different chemical polarity.

1.2 This method is restricted to use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 REFERENCES

2.1 USEPA, SW-846, 3rd Ed. Method 3630C. December, 1996

3.0 SUMMARY OF METHOD

3.1 This method provides the option of using either standard column chromatography techniques or solid phase extraction techniques.

3.2 Cartridge clean-up protocol uses solid phase extraction cartridges packed with 1 gram of 2 grams of silica gel absorbent. Each cartridge is solvent washed immediately prior to use. Aliquots of sample extracts are loaded onto the cartridges, which are then eluted with suitable solvents. A vacuum manifold is required to obtain reproducible results. The collected fractions may be further concentrated prior to analysis.

4.0 APPARATUS AND MATERIALS

4.1 Silica Gel cartridges 1 gram or 2 grams. (100/200 mesh).

4.2 Vacuum manifold system.

4.3 Pipets – disposable, Pasteur type.

4.4 Collection vials, 20 ml tubes.

4.5 Autosample vials.

4.6 Cyclohexane, pest grade.

4.7 Hexane, pest grade.

4.8 Acetone, pest grade.

4.9 Pentane, pest grade.

4.10 2-Propanol, pest grade.

4.11 Toluene, pest grade.

4.12 Methylene Chloride, pest grade

5.0 PROCEDURE

5.1 Cartridge Conditioning

5.1.1 Arrange the 2 g silica cartridge on the manifold in the closed-valve position. Different gram size of silica cartridges may be used.

5.1.2 Turn on the vacuum pump and set pump vacuum to 10 inches (254 mm) of Hg.

5.1.3 Condition the cartridges by adding 4 ml of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to

remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off vacuum.

5.1.4 Slowly open cartridge valves to allow the hexane to pass through the cartridges. Close the cartridge valves when there is still at leasst 1 mm of solvent above the sorbent bed. If cartridge goes dry, repeat cartridge conditioning steps.

5.2 Handling of Extract

5.2.1 Before the silica gel cleanup technique can be utilized, the extract must be exchanged to hexane and the phenols must have undergone derivatization by pentafluorabenzyl bromide, as per the method.

5.2.2 Use the 2g conditioned silica gel cartridges.

5.2.3 Rinse the cartridge by using two 5 ml aliquots of hexane. Discard aliquots.

5.2.4 Transfer the extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 ml/minute.

5.2.5 When the entire extract has passed through to cartridges, but before the cartridge becomes dry, rinse the sample vials with an additional 5.0 ml of solvent. Add the rinse to the sample collection vial.

5.2.6 Close the cartridge valve and turn off vacuum after the solvent has passed through, ensuring the cartridge never goes dry. Replace the collection vial.

5.2.7 Add 5 ml of toluene/hexane (25/75, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial.

5.2.8 Adjust the final volume of the eluant to a known volume which will result in analyte concentrations appropriate for the project requirements using techniques described in an appropriate 3500 series method. The cleaned extracts are ready for analysis.

6.0 QUALITY CONTROL

6.1 Before any sample can be processed using silica cartridges, the analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentration. The efficiency of each lot of the solid-phase extraction cartridges must be verified. The recovery for each analyte must be greater than 85%. Only lots of cartridges from which the spiked analytes are quantitatively recovered may be used to process the samples. A check should also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

Table 4
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (25ng DFTPP)	Every 12 hours.	Ensure correct mass assignment. DFTPP % Relative abundance criteria as specified in Table 3. Pentachlorophenol tailing < 2, Benzidine tailing < 2 DDT breakdown < 20%	Retune. Do not proceed with analysis until DFTPP spectrum meets criteria.
Initial Calibration	Each time the instrument is set up and when CCCs in the continuing calibration verification (CCV) do not meet criteria. Established initially at minimum five concentration levels (six concentration levels if a second order {quadratic} curve is used) - low standard at or below project required reporting limit (PRRL), near but above method detection limits (MDL).	1. % RSD for RRFs for each CCC ≤30%. 2. % RSD for RRFs for all target compounds ≤15%. IF RF % RSD >15% use linear curve, r >=.99, or quadratic curve, r2 > = .990. (NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria should be followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	Should be at or near the mid-point of calibration range for all target compounds, CCCs, and surrogates and should also be prepared from second source standards.	1. % Deviation. for RRFs of each CCC <20%. 2. Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to one target compound <= 40% RSD. 3. QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria should be followed as stated in their documents (ex: <25%D for QSM projects.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % relative standard deviation (RSD) >20%, then system must be inspected and problem corrected before sample analysis. If >20% RSD then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. QSM allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification standards(CCV)	Every 12 hours. Should be at or near the mid-point of calibration range for all target compounds, CCCs, SPCCs and surrogates and is prepared from standards used for calibration.	1. % Deviation for RRFs of each CCC <20%. 2. Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to one target compound <= 40% RSD. 3. QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria should be follow as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % RSD >20%, then system must be inspected and problem corrected before sample analyses. If >20% RSD correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. Sample results reported that have %D failures must be qualified (Z). QSM allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in CCV level of ICAL. 2. Retention time (RT) within 30 sec of RT for associated CCV standard. 3. QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria should be follow as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Reextract and reanalyze sample. * If reanalysis is outside limits the data should be qualified (S). Follow specified criteria as stated in Shell or other documentation.
Method Blank (MB)	One per prep batch of 20. The MB is used to document contamination resulting in the analytical process and should be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern should be less than the highest of either : Method detection limit, 5% of the regulatory limit for that analyte, or 1/5 of the measured concentration in the sample. 2. QSM ≤ ½ MRL Common lab contaminants < MRL. 3. Follow criteria according to specific program/agency.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If reanalysis of blank still contains contamination above specified limits, affected data should be flagged (B). If method blank is in noncompliance, re-extract and reanalyze blank and samples.* For QSM data if less than ½ MRL no action required.* Apply "B" to all associated positives when less than 5x blank concentration and 10x for common lab contaminants (phthalates).

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per prep batch of 20 samples. Must undergo all sample preparation procedures. Spiking solution should contain all target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries (and RPDs, if applicable) within in-house generated limits. Default 70-130% (20% RPD). Sporadic marginal failure (SMF) of up to 10 % of target compounds = 60-140 % rec. 2. Use QSM, Project Specific or Client limits when applicable.	If LCS recoveries are within control limits or within SMF frequency and limits then no action is required. If the LCS exceeds control limits, as well as SMF criteria the reanalyze the LCS to confirm proper preparation procedure. If sample data is reported with LCS failures then that data must be qualified (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.
MDL Level Verification MDL check (2X MDL)	Once per quarter per instrument	1. Analytes detected	Correct problem and repeat.
MRL Level Verification Check standard at Reporting Limit.	Beginning and End of 12 hr. sequence.	1. 70-130% or project specific/client limits 2. SMF 60-140%.	Run MDL check to verify that compounds can be detected.
Matrix Spike/Matrix Spike Duplicate	One set per prep batch of 20 samples. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	2. % Recoveries and RPDs within in-house generated limits. Default 70-130 / 20%RPD. 2. Use QSM, Project Specific or Client limits when applicable.	If LCS is acceptable, then report probable matrix interference. Qualify data if the recoveries are low (M) If recoveries are high and there are no detects in the unspiked sample then that data does not require flagging. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Qualitative/Quantitative Issues	If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed.	The instrument level of all compounds must be within the calibration range for all samples.	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results should be flagged (X).
Surrogate	1. Calibrated as target compounds. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. All % Recoveries within in-house generated limits. Default 70-130%. 2. Use QSM, Project Specific or Client limits when applicable.	If more than one recovery is not within limits: Check to be sure that there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance. If no problem is found, reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample should be qualified (S).

Table 5
8270 SIM Exp. Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MRL					
b. Linearity.	RSD≤ 15%, ≤ 30% for CCCs, r ≥ 0.995 , r2 > 0.990 for regression.					
c. Were the standards used for the ICAL uniquely identified?						
d. Was there a DFTPP standard analyzed prior to the ICAL?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the CCCs within QC limits	%D ≤ 20%					
c. Were the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs for target analytes analyzed at the beginning of the sequence and after every 12 hours.						

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
b. Were the recoveries for the CCVs acceptable?	%D≤20%,					
c. Was each CCV uniquely identified (i.e. Standard Number)?						
4. DFTPP						
a. Was a DFTPP tune check ran at the beginning of every twelve hour shift?						
b. Were the relative abundance criteria met?						
c. Was the peak tailing < 2 on Pentachlorophenol and < 2 on Benzidine?						
d. Was the breakdown of DDT to DDE and DDD less than 20%						
5. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Were the MB results less than the reporting limit (RL)?	< MRL					
If no, were positive hits in the samples flagged with a "B".						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤ 20 samples					
6. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits					
If no, and the recoveries were low, flag those analytes "Q". If the recoveries were high, only flag the detects (>RL) for those analytes "Q".						
7. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits					
c. Is the relative percent difference (RPD) for each analyte between a matrix spike (MS) and matrix spike duplicate (MSD) within the acceptance criteria? (same criteria for LCS and LCSD)	20% RSD or In-house limits					
9. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extraction?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. GPC, acid-base fractioning, or silica gel treatment)	Acid/Base, GPC, Silica gel					
10. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the data file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

CT Laboratories.

Title: **Ion Chromatography**

SOP Number: **CC-IC**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method covers the sequential determination of the anions chloride, fluoride, nitrite, nitrate, *ortho*-phosphate, and sulfate in all water samples as well as soil and sludge samples that have been extracted according to the procedure in this SOP.

2.0 METHOD SUMMARY

2.1 A small volume of a water or extracted sample is injected into an ion chromatograph to flush and fill a 25 or 50uL constant volume sample loop. The sample is then injected into a stream of carbonate-bicarbonate eluent.

2.2 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a guard column and a separator column, are packed with low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a self-regenerating suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions, in their acid form, are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak area and comparing it to a calibration curve generated from known standards.

3.0 DEFINITIONS

3.1 Calibration Blank (CB)—A volume of reagent water fortified with the same matrix as the calibration standards. It is analyzed immediately following the calibration standards (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration, or possible analyte carry-over. Control criteria consist of the absolute value being less than or equal to the MDL for a given analyte. If this range is exceeded, a new calibration will be necessary or data flagged appropriately.

3.2 Calibration standard (CAL)—A solution prepared from the stock standard solutions. The CAL solutions are used to calibrate the instrument. Acceptance of the calibration requires a correlation coefficient of 0.995 or better. No samples will be analyzed without acceptable calibration.

3.3 Laboratory Control Standard (LCS)-- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is analyzed exactly like a sample and its purpose is to determine whether the methodology is in control. The retention times of the analytes in the LCS must be within 10% of the retention time of the those analytes in the calibration curve.

3.4 Matrix duplicate (DUP)—Two separate aliquots of a sample are analyzed at a rate of one per 10 samples of a given matrix per day for 9056 and 300.0. ACOE duplicates should be analyzed at a rate of one per project per run or as specified by

the client. To be considered acceptable RPD between the sample and the sample duplicate must meet in house acceptance criteria.

3.5 Matrix spike (MS)—An aliquot of a sample to which known quantities of the analytes of interest are added. The MS is analyzed exactly like the samples and its purpose is to determine whether the sample matrix contributes bias to the analytical results. An MS is prepared for every 20 samples of a given matrix per day for 9056 or CLP, for every 10 samples of a given matrix per day for 300.0, and at the same rate as the matrix duplicates for ACOE work. Failure to meet criteria may be due to matrix interference within the sample. To be considered acceptable, MS must meet in house % recovery criteria.

3.6 Matrix spike duplicate (MSD)—An additional aliquot of sample treated exactly as the MS. The MSD is run along with the DUP and MS for ACOE work. To be considered acceptable it must meet the same RPD criteria as the DUP and % recovery criteria as the MS.

3.7 Method blank (MB)—An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the lab environment, the reagents or the apparatus. A minimum of one MB is prepared per batch of 20, and is analyzed at the beginning of an analytical batch. Blank recovery should be less than the MDL.

3.8 Linear calibration range (LR)—the concentration range over which the instrument response is linear. Samples with results greater than the highest calibration standard should be diluted to a concentration that falls within the calibration curve range and reanalyzed.

3.9 Method detection limit (MDL)—The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 Calibration Verification Standard-Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. The ICV must be from a second source different than that of the calibration standards, while the CCV may be from the same source as the calibration standards.

3.11 Contract Required Detection Limit (CRDL) Standard--Detection level standard at a level near the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

4.0 HEALTH AND SAFETY

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonable achievable.

4.2 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

5.1 Nitrate, Nitrite and *o*-Phosphate have 48 hour holding times. Nitrate and nitrite can be analyzed together when they are preserved with sulfuric acid. The combined Nitrate/Nitrite test has a 14 day holding time when preseverd with acid but cannot be used to speciate between Nitrate and Nitrite. Chloride, Sulfate and Fluoride have a 28 day holding time when stored at 4^0C.

5.2 Standards should be stored at 4° C.

6.0 INTERFERENCES

6.1 Any species with a retention time similar to that of the desired ion will interfere. Large quantities of ions eluting close to the ion of interest will also result in an interference. Separation can be improved by adjusting the eluent concentration and/or flow rate. Sample dilution and/or the use of the method of standard additions can also be used. Two common species, formate and acetate, elute between fluoride and chloride.

6.2 Because bromide and nitrate elute very close together, they are potential interferences for each other. It is advisable not to have Br^- /$N0_3^-$ ratios higher than 1:10 or 10:1 if both anions are to be quantified.

6.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms. Insure that all glassware is rinsed with Milli-Q water prior to use and use only Milli-Q water for making standards and reagents.

6.4 Samples that contain particles larger than 0.45 µm and reagent solutions that contain particles larger than 0.20 µm require filtration to prevent damage to instrument columns and flow systems.

6.5 Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution can be used to solve most interference problems associated with retention times.

6.6 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak. This is taken care of by using the void volume function of the software for the fluoride peak.

6.7 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant.

6.8 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

7.0 PERSONNEL QUALIFICATIONS

Personnel operating the IC should have background knowledge of the scientific principles used during this application. All operators should perform an initial demonstration of capability (IDC) prior to analyzing any samples. It is preferable for the operator to have at least two semesters of college chemistry.

8.0 APPARATUS AND MATERIALS

8.1 Dionex DX-120 Ion chromatograph

8.1.1 Guard column, Dionex AG14A-7uM, placed before the separator column to protect the separator column from being fouled by particulates or certain organic constituents (4 x 50 mm, Dionex P/N 056897 or equivalent)

8.1.2 Separator column, Dionex AS14A-7uM,a column packed with low-capacity pellicular anion exchange resin that is etheylvinylbenzene crossed with divinylbenzene (4 x 250 mm, Dionex P/N 056904 or equivalent)

8.1.3 Self-regenerating suppressor, Dionex SRS-Ultra, a column that is capable of converting the eluent and separated anions to their respective acid forms (Dionex P/N 53946 or equivalent)

8.1.4 Detector, a low-volume, flow-through, temperature-compensated, electrical conductivity cell (approximately 1.25 µL volume, Dionex DS4 or equivalent) equipped with a meter capable of reading from 0 to1,000 µseconds/cm on a linear scale.

8.1.5 Pump, capable of delivering a constant flow of 0.5 to 4 mL/min throughout the test.

8.1.6 Autosampler, Dionex AS40 with autosampler trays (Dionex P/N 046032 or equivalent), tubes and filter tops (Dionex P/N 038141 or equivalent).

8.1.7 Eluent reservoirs, suitable containers for storing eluent under pressure.

8.1.8 Computer with PeakNet 5.1 software and a printer, to integrate the area under the chromatogram. Software controls the DX-120 as well as processing and calculating data for the DX-120. Printer generates a paper copy of the data for archival purposes.

8.1.9 Bed support assembly- (Dionex P/N 042955 044689 or equivalent)

8.2 Analytical balance, capable of weighing to the nearest 0.0001 g.

8.3 Pipets, Class A volumetric flasks, beakers: assorted sizes

8.4 Autopipettors of various volumes (Oxford Macro and Eppendorf micro)

8.5 Syringes

8.6 Glass Fiber Syringe Pre-Filters

8.7 0.45um Syringe filters with pre-filter

8.8 Reagents

8.8.1 Reagent water--Milli-Q water

8.8.2 Eluent Concentrate—Into a 2L volumetric flask add 84.0g dried sodium bicarbonate and 371.0g dried sodium carbonate and bring to volume with Milli-Q water.

8.8.3 Eluent—Use 20mL eluent concentrate (8.8.2) and bring to 10 liters with Milli-Q water in carboy. Helium purge to remove bubbles prior to use. Filter if necessary. Store on counter in IC lab.

8.8.4 Nitrate Nitrogen-200mg/L- Into a 1L volumetric flask, add 1.444g of Potassium Nitrate and 2.0mL of chloroform. Bring to volume with Milli-Q water. Store in standards refrigerator in wet chem lab.

8.8.5 Nitrite Nitrogen-200mg/L- Into a 1L volumetric flask, add 1.214g of Potassium Nitrite and 2.0mL of chloroform. Bring to volume with Milli-Q water. Store in standards refrigerator in wet chem lab.

8.8.6 LCS/ICV/CCV Concentrate #1—Into a 1L volumetric flask add 40mL 100mg/L Flouride, 20mL 1000mg/L Chloride, 26.55mL 226mg/L Nitrogen in the form of Nitrate (alternately use 6.0mL 1000mg/L N as NO3 or 30mL 200mg/L N as NO3(8.8.4)), 4mL 1000 mg/L *ortho*-phosphate and 20mL 1000mg/L Sulfate. Bring to volume with Milli-Q water. Store in the refrigerator in the wet chemistry laboratory. TV = 4mg/L F, 20mg/L Cl, 6mg/L N as NO3, 4mg/L O-Phos and 20mg/L SO4. Expires after one year or sooner if stock solutions expire first.

8.8.7 LCS/ICV/CCV Concentrate #2—Into a 1L volumetric flask add 6mL 1000mg/L Nitrogen in the form of Nitrite (alternately use 30mL 200mg/L N as NO2 (8.8.5)) and fill to volume with Milli-Q water. Store in the refrigerator in the wet chemistry laboratory. TV = 6mg/L N as NO2. Expires after one month.

8.8.8 LCS/ICV/CCV- Into a 100mL beaker add 30mL LCS Concentrate #1 (8.8.6) and 30mL LCS Concentrate #2 (8.8.7). Mix well. Use immediately. True value for solution is Fluoride and Phosphorus 2mg/L, Chloride and Sulfate 10mg/L and Nitrate and Nitrite Nitrogen 3 mg/L. ***NOTE: ACOE requires that the LCS/ICV/CCV be exactly one-half the value of the high calibration standard. Adjust concentrations accordingly.***

8.8.9 100mg/L Chloride--add 10mL of 1000 mg/L Chloride to a final volume of 100mL with Milli-Q water.

8.8.10 100mg/L Nitrite Nitrogen—add 10mL of 1000 mg/L Nitrite Nitrogen (alternately use 50mL of 200mg/L Nitrite Nitrogen) to a final volume of 100mL with Milli-Q water.

8.8.11 100mg/L Nitrate Nitrogen—add 10mL of 1000 mg/L Nitrate Nitrogen (alternately use 50mL of 200mg/L Nitrate Nitrogen or 44.25mL of 226mg/L Nitrate Nitrogen) to a final volume of 100mL with Milli-Q water.

8.8.12 100 mg/L Phosphorus--add 10mL of 1000 mg/L Phosporus to a final volume of 100mL with Milli-Q water.

8.8.13 Calibration standards- Into 8-100mL volumetric flasks pipet the following and fill to volume with Milli-Q water.

Level	1	2	3	4	5	6	7	8
Vol of 100mg/L F	0	0.02	0.10	0.5	1.0	2.0	5.0	7.0mL
Vol of 100mg/L Cl	0	0.04	0.50	1.0	5.0	10.0	20.0	30.0mL
Vol of 100mg/L NO2	0	0.02	0.10	0.5	1.0	2.0	5.0	7.0mL
Vol of 100mg/L NO3	0	0.02	0.10	0.5	1.0	2.0	5.0	7.0mL
Vol of 100mg/L P	0	0.02	0.10	0.5	1.0	2.0	5.0	7.0mL
Vol of 1000mg/L SO4	0	0.025	0.05	0.1	0.5	1.0	2.0	5.0mL

Prepare fresh daily. **Calibration Standards must be made from different sources than the LCS.**

8.8.14 Calibration check concentrate #1—Prepared as the LCS/ICV/CCV Concentrate #1 (8.8.6) with standards from the same source as the calibration. Store in the wet chemistry refrigerator.

8.8.15 Calibration check concentrate #2—Prepared as the LCS/ICV/CCV Concentrate #2 (8.8.7) with the same standard used to make the calibration. Store in the wet chemistry refrigerator.

8.8.16 Calibration Check—Into a portion cup combine 5.0mL of Calibration Check Concentrate #1 (8.8.15) and 5.0mL of Calibration Concentrate Concentrate #2 (8.8.16). Mix well. Prepare fresh daily.

8.8.17 Spiking solution #1- Into a 200mL volumetric flask pipet 64mL 100mg/L F, 51.2mL 1000mg/L Cl, 12.8mL 1000mg/L N as NO3 (alternately 60.64mL 200mg/L N as NO3 or 56.64mL 226mg/l N as NO3), 6.4mL 1000mg/L P and 51.2mL 1000mg/L SO4. Bring to volume with Milli-Q water. True value is 256mg/L Cl and SO4, 64mg/L N as NO3 and 32mg/L F and P. Add 0.1mL to 3.0mL of sample for a true value of 8mg/L Cl and SO4, 2mg/L N as NO3 and 1mg/L F and P. Store solution in wet chem refrigerator. Expires after one year.

8.8.18 Spiking solution #2- Into a 200mL volumetric flask pipet 12.8mL 1000mg/L N as NO2 (alternately 60.64mL 200mg/L N as NO2). Bring to volume with Milli-Q water. True value is 64mg/L N as NO2 . Add 0.1mL to 3.0mL of sample for a true value of 2mg/L N as NO2. Store solution in wet chem refrigerator. Expires after one month.

8.8.19 Sodium Chloride (NaCl)- 1M- Dissolve 58.5g of NaCL in DI water in a 1 Liter volumetric flask and dilute to volume with DI water. Adjust the pH to 2.0 using HCl. Stored in plastic bottle on top of IC.

8.8.20 pH 2.0 200 mM NaCl in 80% acetonitrile- Into a 500mL volumetric flask, combine 100mL of 1M NaCl solution (8.8.12) and 400mL of Acetonitrile (stored in extraction lab). This solution needs to be made fresh for each use.

8.8.21 10X Eluent--Into a 500mL volumetric flask, add 10.0mL of Eluent Concentrate (8.8.2) and dilute to volume with Milli-Q water.
8.8.22 Oxalic Acid solution- 0.1 M
8.8.23 200mN sulfuric acid- Into a 1 liter volumetric flask, add 5.6 mLs of concentrated sulfuric acid and dilute to volume with DI water.

9.0 INSTRUMENT CALIBRATION

9.1 Start DX-120 and allow to equilibrate. See section 11.0 for further instructions on start up and method development.

9.2 In the PeakNet software, go to the Method Editor. Open a new file. See Section 11.3 for further instructions on method set-up.

9.2.1 Click on the Calibration Parameters icon.

9.2.1.1 Choose the General page. Choose the method of standardization to be external, the number of replicates is 1 the linear weighting is equal and the calibration standard volume is 1. Check the Replace Retention Time and Update Response/replace under the Update Data.

9.2.1.2 Choose the Defaults page. The sample volume, sample weight and dilution factor should all default to 1. The unknown response factor should default to 0 and the response for unknowns should default to area.

9.2.2 Click on the Component table icon.

9.2.2.1 On the Identification page:

9.2.2.1.1 The components for the anions group will elute in the following order: Fluoride, Chloride, Nitrite, Nitrate, *ortho*-Phosphate and Sulfate.

9.2.2.1.2 The retention times will be set based on the elution time of the analytes in a known standard. See method set up in Section 11.0 for further instructions.

9.2.2.1.3 For the Tolerance, choose 0.5 minutes.

9.2.2.1.4 For the Reference Component, choose none.

9.2.2.2 On the calibration Standards page:

9.2.2.2.1 The concentrations for each level in mg/L are:

Level	1	2	3	4	5	6	7	8
F	0	0.02	0.1	0.5	1	2	5	7
Cl	0	0.04	0.5	1	5	10	20	30
NO_2	0	0.02	0.1	0.5	1	2	5	7
NO_3	0	0.02	0.1	0.5	1	2	5	7
o-Phos	0	0.02	0.1	0.5	1	2	5	7
SO_4	0	0.25	0.5	1	5	10	20	50

9.2.2.3 On the Calibration page:

9.2.2.3.1 For the curve fit type, choose linear.

9.2.2.3.2 For the Origin, choose ignore

9.2.2.3.3 For Calibrate by, choose Area.

9.2.3 Save the method under C:\Instrument Archive\Method\Sytstem x\W####, where x is the system number and #### is the calibration standard number for the calibration curve.

9.3 Go to the Schedule Editor.

9.3.1 Under sample name, enter Level number. Repeat this for all eight levels.

9.3.2 Under the Sample type, choose calibration standard

9.3.3 Under Level, choose the appropriate level number for each level

9.3.4 Under the method name, choose the method just created (C:\instrument archive\method\system x\W####)

9.3.5 Under data file copy and paste the entries from the sample name column.

9.3.6 Save schedule under c:\instrument archive\schedule\system *\W####

9.4 Go to the Run Menu.

9.4.1 Go to file and choose load schedule.

9.4.1.1 Choose the schedule that you saved under c:\instrument archive\schedule\system *\W####

9.4.2 On the Sample page:

9.4.2.1 Under Data File, choose Browse.

9.4.2.2 Create a new directory under c:\instrument archive\data\system *. Name the new directory by the calibration standard number. Click OK

9.4.2.3 Under Data Collection, confirm that the defaults of DX-120 and the method run time appear.

9.4.3 On the Modes page:

9.4.3.1 Choose upon receiving signal at module.

9.5 On the AS-40 Autosampler:

9.5.1 Load the calibration standards in order.

9.5.2 Press the Run button to begin running.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 For Nitrate, Nitrite and *o*-Phosphate, refrigerate water and soil samples at 4^0C until analyzed. Hold time for samples is 48 hours.

10.2 For Chloride, Sulfate, and Fluoride, refrigerate water and soil samples at 4^0C until analyzed. Hold time for samples is 28 days.

10.3 For Nitrate + Nitrite, preserve samples to a pH <2 with sulfuric acid. Refrigerate samples at 4^0C until analyzed. Hold time for samples is 14 days.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Sample Preparation

11.1.1 For water samples, filter, if necessary, with a 0.45um syringe filter 3-5mL of an appropriately preserved sample into an autosampler vial and cap with a filter cap.

11.1.2 For soil samples, place 5g $\pm$0.01g of sample in a specimen cup and add 50mL of Milli-Q water. Place on shaker table for 30 minutes then filter 3-5 mL of sample through a 0.45um syringe filter into an autosampler vial and cap with a filter cap.

11.2 Instrument equilibration

11.2.1 Turn on DX-120.
11.2.2 Ensure that Helium tank has an adequate supply of Helium for the run.
11.2.3 Fill the eluent containers on the top of the instrument with degassed eluent.
11.2.4 Turn on the Helium supply for the DX-120.
11.2.5 Press the Local/Remote button on the front of the DX-120. Make sure that the instrument screen on the front of the DX-120 says Local.
11.2.6 Press the Eluent pressure button. A green light should indicate that it is on.
11.2.7 Open the front cover of the instrument and locate the small screw, in the middle of the panel, that controls the purge for the pump.
11.2.8 Loosen the screw by turning 2 or 3 times
11.2.9 Press the pump button on the front of the instrument. A green light should indicate that it is on.
11.2.10 Allow the pump to prime for approximately one minute. If air is not purged from the system, increase the flow rate by pulling the knob adjacent to the pump and turning it clockwise. Turn the knob until the air is purged or until the flow rate is approximately 2.0mL/min. Allow the pump the prime for approximately one minute.
11.2.11 Close the pump screw
11.2.12 Press the SRS button on the front of the instrument. A green light should indicate that it is on. If the flow rate was increased to prime the pump return it to its original setting by turning the knob counterclockwise.
11.2.13 Allow instrument to stabilize for 20-30 minutes prior to analysis.

11.3 Method creation in PeakNet software
11.3.1 Turn on computer
11.3.2 Open the PeakNet software by double clicking on the icon
11.3.3 Open the Method editor by left clicking on the Method icon.
11.3.4 Click on the new file icon (blank sheet) in the toolbar to open a new method file.
11.3.5 Highlight DX-120 under modules and then click add. Press Exit.
11.3.6 After the dialog box opens
11.3.6.1 Set the data collection time to 15 minutes.
11.3.6.2 Set the rate to 5.00 Hz
11.3.6.3 Set the Detector Unit to uS
11.3.6.4 Set the Plot Scales to 30.0 uS for the maximum and –3uS for the minimum.
11.3.6.5 Click on the timed events icon (stopwatch).
11.3.6.5.1 For time INIT, place a check mark next to TTL1. All others should be unchecked. Click enter.
11.3.6.5.2 For time 0.0, place a check mark next to offset. All others should be unchecked. Click enter.
11.3.6.5.3 For time 0.01, place a check mark next to begin. All others should be unchecked. Click enter.
11.3.6.5.4 Click exit.
11.3.6.6 Click on the integration icon (peak).
11.3.6.6.1 Set the Peak detection algorithm to standard
11.3.6.6.2 Set the peak width to 10.00 seconds.
11.3.6.6.3 Set the peak threshold to 0.01

11.3.6.6.4 Set the area reject to 100
11.3.6.6.5 Set the reference area reject to 100
11.3.6.6.6 Click OK
11.3.6.7 Click on the smoothing parameters icon.
11.3.6.7.1 Set the filter type to none.
11.3.6.8 Click on the data events icon (baseline)
11.3.6.8.1 Move the time line to the time that corresponds to the elution time of Fluoride (usually around 3 minutes).
11.3.6.8.2 Highlight the event option, void volume treatment for this peak.
11.3.6.8.3 Click add event
11.3.6.8.4 Click exit
11.3.6.9 For the calibration and component table icons, refer to section 9.0 to determine the correct settings.
11.3.6.10 Save the method as C:\INSTRUMENT ARCHIVE\METHOD\ SYSTEMx\ W####where x = the system number the method is for and the #### is for the W number of the standard prep.
11.3.6.11 Close the method editor.
11.3.6.12 NOTE: a calibration must be run for the method before it can be used to analyze samples. See Section 9.0 for further details.

11.4 Creating a schedule
11.4.1 Open the schedule editor by clicking on the icon.
11.4.2 A new file should appear. If not, click on the new file icon in the toolbar.
11.4.3 Under the sample column
11.4.3.1 Type in the CTI sample ID for the sample to be analyzed. For DUP, MS and MSD samples, place the letters DUP, MS or MSD in front of the CTI sample ID.
11.4.3.2 The first line in the schedule editor should be the CAL CHECK.
11.4.3.3 The second line in the schedule editor should be ICV.
11.4.3.4 The third line in the schedule editor should be ICB.
11.4.3.5 After every ten samples and at the end of the run, a CCV, which is also used as an LCS, and CCB, which is also used as a MB, should be run.
11.4.4 Under the Method column, all lines should haveC:\ INSTRUMENT ARCHIVE\Method\System x\ W####.met, where W#### is the calibration standard number and x is the system number you are using.
11.4.5 Under the data file column, enter the same thing as the sample field. It is easier to copy and paste the sample column for this rather than hand entering all the information again.
11.4.6 Under the comments column, enter any dilution factors that you might have.
11.4.7 Save the schedule as c:\ INSTRUMENT ARCHIVE\Sequences\System X****** where x is the system number you are using and ****** is the LIMS run number.
11.4.8 Exit the schedule editor.

11.5 The run program in PeakNet
11.5.1 Click on the Run icon (center of screen)
11.5.2 Click on the window for the system you want to use.
11.5.3 Click on file. Choose Load Schedule.

Select the schedule for that corresponds to the LIMS run number you want to run.

11.5.4 Click Open

11.5.5 Under data on the load schedule page

11.5.5.1 Click on browse

11.5.5.2 Double click on the folder under C:\instrument archive\data\system x

11.5.5.3 In the new directory box type in the LIMS run number that you want to run.

11.5.5.4 Click Create to create the file folder

11.5.5.5 Click OK to get back to the load schedule page

11.5.6 Under modes on the load schedule page

11.5.6.1 Click on the "upon receiving signal at module" button.

11.5.7 Click OK at the bottom of the Load Schedule page. The computer will be waiting for the autosampler to signal that a sample has been injected.

11.6 Load the autosampler with the samples in the order indicated in the schedule editor.

11.7 Press the Run/Hold button on the Autosampler. A green light will indicated that the autosampler is running.

11.8 To turn off the DX-120

11.8.1 Close the run in PeakNet

11.8.2 Close PeakNet software

11.8.3 On the DX-120:

11.8.3.1 Press the SRS button. The green light next to SRS should shut off.

11.8.3.2 Press the pump button. The green light next to pump should shut off.

11.8.3.3 Press the eluent pressure button. The green light next to the eluent pressure should shut off.

11.9 If system will remain off for an extended period of time

11.9.1 Press the button on the front of the DX-120 to turn off the power to it.

11.9.2 Turn off the power to the autosampler using the button on the left rear.

12.0 DATA CAPTURE

12.1 Select Batch in the PeakNet Main Menu.

12.2 Under File click open

12.2.1 Open C:\PeakNet\Schedule\DC.bch

12.3 Under Processing select Input

12.3.1 Click Select

12.3.1.1 Select the schedule you want to data capture. C:\Insturment Archive\ Schedule\System x***** where ***** is the LIMS run number.

12.3.1.2 Click Open

12.3.2 Process Injection should default to 1 through the last line of your schedule. If there are more than 100 lines in the schedule capture it in parts. For example when capturing a schedule that has 129 lines change the 129 to 100. Capture the schedule then repeat the process changing the 1 to 101 and leaving the 129.

12.3.3 Select All under included Detectors

12.3.4 Choose from schedule for process method

12.4 Under Output unselect all options.

12.5 Select Export

12.5.1 Change the file name to I:\DX120***** +.csv where ***** is the LIMS run number followed by +, an arbitrary letter. If capturing more than once to the same run, changing this letter prevents the files from being overwritten.
12.5.2 Select summary as the report type.
12.5.3 Choose Peak as the summary option.
12.6 Click OK
12.7 Click the Start Button
12.8 Exit the batch program. Do not save the changes.

13.0 TROUBLESHOOTING AND MAINTENANCE

13.1 See Dionex PeakNet Software User's Guide, Dionex DX-120 Operator's manual, Dionex Installation Instructions, Troubleshooting Guide for the IonPac AS14, Column Care Guide for IonPac AS14 and ASRS-Ultra Installation Instructions and Troubleshooting Guide for troubleshooting instructions not covered in this section.

13.2 The column bed support assemblies should be replaced when they become clogged. A clog usually causes an increase in the pressure of the system. NOTE Replace the outlet bed support ONLY if high pressure persists after replacement of the inlet fitting.

13.2.1 Disconnect the column from the system.
13.2.2 Carefully unscrew the inlet (top) column fitting. Use two open-end wrenches.
13.2.3 Remove the bed support. Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that you DO NOT SCRATCH THE WALLS OF THE END FITTING. Discard the old bed support assembly.
13.2.4 Place a new bed support assembly into the end fitting. Make sure that the end of the column tube is clean and free of any particulate matter so that it will properly seal against the bed support assembly. Use the end of the column to carefully start the bed support assembly into the end fitting. If the column tube end is not clean when inserted into the end fitting, particulate matter may obstruct a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.
13.2.5 Screw the end fitting back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.
13.2.6 Reconnect the column to the system and resume operation.

13.3 The column will need to be cleaned when the retention time window shift more than 10%, calibrations are no longer linear or the column becomes contaminated from a sample.

13.3.1 Prepare a 500 mL solution of pH 2.0 200 mM NaCl in 80% acetonitrile

13.3.2 Disconnect the ASRS-Ultra from the IonPac AS14 Analytical Column. Reverse the order of the guard and analytical column in the eluent flow path. Double check that the eluent flows in the direction designated on each of the column labels.CAUTION When cleaning an analytical column and a guard column in series, ensure that the guard column is placed after the analytical column in the eluent flow path. Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately.

13.3.3 Set the pump flow rate to 1.0 mL/min

13.3.4 Rinse the column for 15 minutes with deionized water before pumping the NaCl/Acetonitrile cleanup solution over the column.

13.3.5 Pump the NaCl/Acetonitrile cleanup solution through the column for 60 minutes.

13.3.6 Rinse the column for 15 minutes with deionized water before pumping the Oxalic acid cleanup solution over the column.

13.3.7 Pump the Oxalic Acid cleanup solution through the column for 60 minutes.

13.3.8 Rinse the column for 15 minutes with deionized water before pumping the 10X eluent cleanup solution over the column.

13.3.9 Pump the 10X eluent cleanup solution through the column for 60 minutes.

13.3.10 Rinse the column for 15 minutes with deionized water before pumping eluent over the column.

13.3.11 Equilibrate the column(s) with eluent before resuming normal operation for at least 30 minutes.

13.3.12 Reconnect the ASRS-Ultra to the AS14 Analytical Column and place the guard column in line between the injection valve and the analytical column.

13.4 Small analyte peak area is caused by running eluent through the ASRS while the power is off to the ASRS. To regenerate the ASRS:

13.4.1 Disconnect the eluent line from the analytical column attached to the ELUENT IN port of the ASRS-ULTRA at the analytical column end of the line. Direct this line to a separate waste beaker.

13.4.2 Disconnect the eluent line from the ELUENT OUT port of the ASRS-ULTRA.

13.4.3 Using a plastic syringe push approximately 3 mL of 200 mN sulfuric acid through the ELUENT OUT port and approximately 5 mL of 200 mN sulfuric acid through the REGEN IN port.

13.4.4 Reconnect the eluent line from the ELUENT IN port of the ASRS-ULTRA to the analytical column and the eluent line from the **ELUENT OUT** port of the ASRS-ULTRA to the conductivity detector cell.

13.4.5 Turn on the power and then begin pumping eluent.

14.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

13.1 LIMS will calculate the following according to these equations:

13.1.1 *Liquid Concentration (mg/L)* = A x C
where A = instrument reading for sample (mg/L)
C= dilution factor

13.1.2 *Solid Concentrations (mg/kg)* = $\frac{A \times B \times C}{D \times E}$,
where A = instrument reading for sample (mg/L)
B = total volume of extraction (L)
C= dilution factor
D = amount of sample used in extraction (Kg)
E = percent solids/100 (if reporting on a Dry Weight Basis)

13.1.3 *Spike Recovery (%)*= $\frac{\text{(Spiked sample value – Sample value) x 100}}{\text{(Spike amount)}}$

13.1.4 *%RSD* = $\frac{|\text{(Sample – DUP)}| \times 100}{\text{(Sample + DUP)/2}}$,
where DUP = Duplicate concentration

15.0 COMPUTER HARDWARE AND SOFTWARE

15.1 Computer with LIMS
15.2 Printer
15.3 PeakNet 5.0 Software

16.0 DATA MANAGEMENT AND RECORD MANAGEMENT

16.1 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.
16.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
16.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.
16.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

17.0 QUALITY CONTROL AND QUALITY ASSURANCE

17.1 After every 10 injections, analyze a CCV, a midrange calibration standard. If the instrument retention time has changed by more than 10% or recovery is outside of 90-110%, remake solution and analyze the fresh solution. If it still does not fall within the above criteria, recalibrate.
17.2 Analyze one in every ten samples in duplicate for 300.0 or every twenty samples in duplicate for 9056A. Take the duplicate sample through the entire sample preparation and analytical process.
17.3 A matrix spiked sample should be run for each analytical batch or twenty samples for 9056A or 10 samples for 300.0, whatever is more frequent, to determine matrix effects.
17.4 Prior to analyzing samples, each analyst must perform an IDC, initial demonstration of capability. The IDC consists of running a standard solution in quadruplicate and getting a recovery and RPD within method limits.

17.5 Method Detection Limit (MDL)—MDLs must be established for all analytes, using reagent water fortified at a concentration of 3-5 times the estimated instrument detection limit. To determine MDL values, take seven or eight replicate aliquots of the fortified reagent water and process through the entire analytical method. Calculate the MDL using the spreadsheet in H:\MDLs\classical\MDL water blank workbook.xls for waters of h:\MDLs\classical\MDL soil blank workbook.xls for soils. MDLs should be determined annually or whenever there is a significant change in background or instrument response.

17.6 Calibration check standard—A mid-point calibration standard run as a sample. If the instrument retention time has changed by more than 10% or recovery is outside of 90-110%, remake solution and analyze the fresh solution. If it still does not fall within the above criteria, recalibrate. A calibration check standard should be run once a day.

17.7 Calibration Blank (CB)—A volume of reagent water fortified with the same matrix as the calibration standards. It is analyzed immediately following the calibration standards (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration, or possible analyte carry-over. Control criteria consist of the absolute value being less than or equal to the MDL for a given analyte. If this range is exceeded, a new calibration will be necessary or data flagged appropriately.

Standard Quality Control Requirements and Corrective Action Guidelines

QC Type	**Frequency**	**Conc. Level**	*Acceptance Criteria*	*Corrective Action*
ICAL	As needed		R $\geq$ 0.995	Recalibrate Instrument
ICV	1 per calibration	Midpoint of cal curve	90-110%	Reanalyze or recalibrate
ICB	1 per calibration		<MDL	Correct problem and reanalyze or qualify data that is not > 20 times the ICB or <MDL
CCV	1 after every 10th sample	Midpoint of cal curve	90-110%	Correct problem and reanalyze.

CCB	Immediately following each CCV		<MDL	Correct problem and reanalyze or qualify data that is not > 20 times the ICB or <MDL
LCS	1 per batch of 20 samples	Midpoint of cal curve	90-110%	Correct problem and reanalyze.
DUP	10% (1 for every 10 samples) per matrix		In house limits. Default is 20%	Identify source of problem. Qualify data or reanalyze depending on source of problem.
MS-MSD	5% (1 in 20) of samples per batch per matrix		In-house limits. Default limits are 80-120%	Identify source of problem. Qualify data or reanalyze depending on source of problem.

Data Validation Checklist

LIMS #:	Method: Anions by Ion Chromatography 300.0/9056			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Were the samples analyzed within hold time?	28 days for Cl, F, SO_4;14 days for preserved NO_2+NO_3; 48 hours for O.Phos, and unpreserved NO_2 and NO_3					
2. Was the calibration performed using the required number of standards?	Three plus a blank					
3. Is the standard prep log number noted on the analytical report?	---					
4. Was the correlation coefficient acceptable?	>= 0.995					
5. Was Calibration Check standard run prior to analysis of samples?	---					
6. Was the Calibration Check standard recovery acceptable?	90 – 110 %					
7. Were the ICV and ICB run immediately after the calibration check standard?	---					
8. Was the ICV recovery acceptable?	90 – 110 %					
9. Was the ICB result acceptable?	< LOD					
11. Were the CCV's and the CCB's analyzed at the required frequency?	1per 10 samples					
12. Were the CCV recoveries acceptable?	90 – 110 %					
13. Were the CCB results acceptable?	< LOD					
14. Was a LCS run at the required frequency?	1 per 20 samples					
15. Was the LCS recovery acceptable?	90-110 %					

16. Was the LCS used before the indicated expiration date?	---					
17. Were the MS and MSD prepared at the required frequency?	1per 20 field samples					
18. Were the MS and MSD recoveries acceptable?	Within in house QC limits					
Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
19. Was the RPD between the MS and MSD acceptable?	Within in house QC limits					
20. Were all chromatograms checked to ensure accurate peak identification?	---					
21. Are all samples on the job lists accounted for?	---					

CT Laboratories
Baraboo Laboratory Divison

Title: Total and Amenable Cyanide

SOP Number: CC-1

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance *Date*

_____
Laboratory Director *Date*

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of cyanide in drinking, ground, surface and saline waters, domestic, industrial wastes, soil and sediment. The working range is approximately 5-200 ug/L or 5-200 mg/kg. Samples greater than that may be diluted into the working range.

1.2 The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide and cyanide amenable to chlorination.

2.0 METHOD SUMMARY

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

2.2 In the automated colorimetric measurement, the cyanide is converted to cyanogen chloride (CnCl) by reaction with Chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The concentration of NaOH must be the same in the standards, the scrubber solutions, and any dilution of the original scrubber solution to obtain colors of comparable intensity.

3.0 DEFINITIONS

3.1 Initial Calibration Verification (ICV): A midrange check standard analyzed after the calibration has been completed, to evaluate calibration curve and or instrument performance. The ICV is non-distilled and from a different source than the calibration standards.

3.2 Lab Control Sample (LCS): A known concentration obtained from a second source.

3.3 Continuing Calibration Verification (CCV): A check standard analyzed after every ten samples and at the end of the analytical run, to evaluate instrument performance.

3.4 Matrix spike-matrix spike duplicate (MS-MSD): To two of three aliquots of a given sample a known amount of spike solution is added. Matrix spikes and matrix spike duplicates are taken through all steps of preparation and analysis, exactly like a sample. The amount of spike recovered helps to assess the effect of the sample matrix on the analysis. The precision of the method is also determined, by calculating the relative percent difference (RPD) (10.3) of the two spiked aliquots. An MS-MSD should be prepared for every 20 samples in each matrix; an MS-MSD is prepared for every 10 samples when samples are being analyzed according to the Department of Defense-Quality Systems Manual (QSM).

3.5 Method Blank (MB): Reagent water treated exactly as a sample, including exposure to all glassware, reagents, and treatments used with other samples.

3.6 Low Distillation Standard (LDS): Low level calibration standard distilled to check low end efficiency of calibration.

3.7 High Distillation Standard (HDS): High level calibration standard distilled to check high end efficiency of calibration.

3.8 Duplicate (Dup): A sample prepared a second time for precision assesment; a QSM/LCG requirement.

3.9 Batch- A batch consists of 20 samples of the same matrix analyzed on the same day or 20 samples of the same medium that have been prepared together.

4.0 HEALTH AND SAFETY

4.1 Cyanogen chloride may form during this reaction. This is a very toxic gas and the distillation should always be performed in a hood.

4.2 Caution should be used during the distillation step. If the vacuum pressure is not adequately maintained, sulfuric acid or sodium hydroxide could be expelled from the distillation apparatus.

4.3 Caution: KCN is highly toxic. Avoid inhalation of dust or contact with the solid or solutions.

5.0 INTERFERENCES

5.1 Chlorine: Oxidizing agents, such as chlorine, decompose most of the cyanides. To determine if there is significant chlorine present, test a drop of the sample with potassium-iodide starch paper. If the paper turns blue, treatment with ascorbic acid will be necessary prior to analysis. Add a few crystals of ascorbic acid a time, until a drop of sample produces no color on the indicator paper; then add an additional 0.6 g of ascorbic acid for each liter of sample.

5.2 Sulfides: Test for presence of sulfide by placing a drop of sample on lead acetate test paper. Darkening of paper indicates presence of sulfide. Treat 75 mL of the reserved sample (pH>12) as follows: Add powdered Cadmium Carbonate to sample gradually. Yellow cadmium sulfide precipitates if the sample contains sulfide. Test the treated sample periodically by adding a drop of the solution to the lead acetate paper. When the paper does not darken, the sulfide has been removed sufficiently. Filter the treated solution through a dry filter paper into a dry beaker. From this filtrate measure out the sample aliquot to be used for analysis. Note any treatment in the sample prep logbook. Avoid a large excess of cadmium and a long contact time.

5.3 NO_3/NO_2: Remove by adding excess (0.2 g) sulfamic acid before distillation.

5.4 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic

acid (1.6M) to pH 6.0 to 7.0. **CAUTION: This procedure can produce lethal HCN gas.** Extract with hexane with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of thesample to above 12 with 50% NaOH solution.

5.5 Most interferences are eliminated reduced by the distillation procedure.

6.0 PERSONNEL QUALIFICATIONS

6.1 Analysts should be familiar with safety requirements for the lab.
6.2 Analysts should have a working knowledge of the midi-distillation system.
6.3 Analysts should have a working knowledge of the Lachat 8000 system.
6.4 Analysts should have a valid IDC on file.

7.0 APPARATUS AND MATERIALS

7.1 Materials
- 7.1.1 Fisher Analytical Balance: To weigh accurately to the nearest 0.0001 g.
- 7.1.2 Glassware: Class A volumetric flasks and pipets.
- 7.1.3 Midi-dist Apparatus: Easy-Dist model, with timed controlled heating block, vacuum controls, digestion tubes, and cold finger condensers, gas scrubbers.
- 7.1.4 Lachat Continuous Flow Autoanalyzer:
  - 7.1.4.1 Sampler
  - 7.1.4.2 Pump
  - 7.1.4.3 Injection Module: 150 cm, 0.8 mm i.d. sample loops
  - 7.1.4.4 Colorimetric Detector with 10mm-flow cell, and 570nm-wavelength interference filter.
  - 7.1.4.5 Heating unit: Temperature must be 60° C.
  - 7.1.4.6 Reaction Module for Cyanide, 10-204-00-1-A
  - 7.1.4.7 QuikCalc II software.
- 7.1.5 Twistcap plastic centrifuge tubes. Stored in wet lab beneath bench
- 7.1.6 PTFE boiling stone. Stored in drawer with CN glasware

7.2 Reagents
- 7.2.1 All reagents are prepared with deionized water.
- 7.2.2 To prevent bubble formation, all solutions except the standards are degassed with helium.
- 7.2.3 Potassium iodide test paper: For checking for chlorine residual and to be used in preparing cyanides amenable to chlorination. Stored on reagent shelf in wet lab.
- 7.2.4 Lead acetate test paper: Used in checking for potential sulfide interference. Stored on reagent shelf in wet lab.
- 7.2.5 Ascorbic Acid: Crystal. Stored on reagent shelf in wet lab.
- 7.2.6 Chloramine T: Dissolve 1.0 g in 50 mL of reagent water in a 250mL volumetric flask. Bring to volume with DI water. Prepare

fresh daily. Do not keep solid reagent for longer than one year. Stored on reagent shelf in wet lab.

7.2.7 Magnesium Chloride Solution: To a 1000-mL volumetric flask, add 500 mL of reagent water. Add 510 g of MgCl2-6H2O (stored on shelf in wet lab) and dilute to 1000 mL with reagent water. Store in plastic container on shelf under hood.

7.2.8 Pyridine-Barbituric Acid Reagent: In a fume hood, place 15.0 g of barbituric acid (stored on reagent shelf in wet lab) in a 1L beaker. Wash the sides of the beaker with about 100-mL reagent water to wet the barbituric acid. Add 75 mL of pyridine (stored under hood in extraction lab) and mix. Add 15-mL conc. Hydrochloric acid (stored under hood in glassware room) and mix. Mix until all the barbituric acid is dissolved. Transfer the solution to a 1L volumetric flask and dilute to the mark with reagent water. Stable for one week if kept in amber bottle in the refrigerator. **Safety Note: Barbituric acid comes in a fine powder form. When weighing this reagent, be sure to take precautions to avoid breathing the powder while weighing the reagent or adding to the beaker.** Bring to room temperature prior to analysis

7.2.9 Potassium dihydrogen phosphate (KH_2PO_4), also know as Potassium phosphate monobasic, buffer: 1M: Dissolve 97 g of anhydrous KH_2PO_4 (stored on reagent shelf in wet lab)in 500mL reagent water in a 1L flask. Stir with a magnetic stirrer for 30 minutes. Bring to volume with DI water Prepare fresh monthly and keep refrigerated. Bring to room temperature prior to analysis.

7.2.10 Sodium Hydroxide Solution, 0.25 M: Into a large carboy, dissolve 80 g of NaOH (stored under hood in wet lab) pellets in reagent water. Bring to 8L with DI water. Prepare as needed and store in caustic cabinet under hood in wetchem lab.

7.2.11 Sodium Thiosulfate, ($Na_2S_2O_3$) as crystals. (Stored on reagent shelf in wet lab)

7.2.12 Standardized Silver Nitrate Solution, 0.0192 N: NIST traceable. (stored under the hood in the extraction lab.)

7.2.13 Indicator solution: 0.02% p-dimethylaminobenzalrhodanine w/v in acetone.

7.2.14 Stock Cyanide Solution (1000 mg /L): Ultra Scientific- Cat # ICC-008 (or equivalent) used for CCVs and calibration. Second Source Std = Lab Chem-Cat. # LC13545-7 (or equivalent), NIST traceable; used for ICVs and spiking. (Keep stock standards refrigerated in the dark). Alternatively the stock standard can be prepared in the lab by dissolving 2.5g KOH and 2.51g KCN in 1L reagent water. Standardize before use and biweekly thereafter.

7.2.14.1 To standardize the 1000 mg/L CN stock standard, fill buret with 0.0192N $AgNO_3$ solution and zero. With a Class A volumetric pipet, pipet 15 mL of stock standard into a 250 mL beaker and into another beaker, pipet 20

mLs of stock standard. To both beakers, add approximately 4 drops of indicator solution (6.13) and place a magnetic stirrer in each. The initial color, when indicator is added, should be a canary yellow. The indicator turns to a salmon hue when the end point is reached. With burette zeroed, begin dispensing the 0.0192N $AgNO_3$ solution into the beaker containing either 15 or 20 mLs of standard. The beaker with the standard should be stirring continuously while dispensing the 0.0192N $AgNO_3$. When the end point is reached (the standard turns from yellow to salmon), take reading off of the burette and record in reagent Standardization Log. Calculate the concentration of the standard using calculation 10.5. An acceptable range for the calculated concentration is 995-1005 mg/L of CN. If the concentration is outside of this range a new stock standard must be made.

7.2.15 Working Stock Standard Solution (10 mg/L)-both sources: Add 1.0 mL of standardized 1000 mg/L CN- stock solution (7.2.14) to a 100 mL volumetric flask. Dilute to the mark with 0.25M NaOH (8.2.10) and mix. Prepare fresh weekly. These standards will be used for spiking and also to prepare working standards.

7.2.16 Working Standards: Prepare fresh weekly (refrigerate and stored in the dark). Using the 10 mg/L working stock solution (7.2.15), transfer volumetrically, into volumetric flasks, the following amounts:

Standard	mLs of	solution into	Vol Flask
200 ug/L	2.0	10 mg/L	100 mLs
100 ug/L	2.0	10 mg/L	200 mLs
50 ug/L	0.5	10 mg/L	100 mLs
30 ug/L	30.0	100 ug/L	100 mLs
20 ug/L	10.0	100 ug/L	50 mLs
10 ug/L	5.0	100 ug/L	50 mLs
0 ug/L	0	---	---

Dilute to volume with 0.25M NaOH and mix well.

7.2.17 Cadmium carbonate: powdered reagent: for reduction of sulfide interference (stored on reagent shelf in wet lab).

7.2.18 Sulfamic Acid: for Nitrate/Nitrite interference: ACS Dry reagent (stored on reagent shelf in wet lab).

7.2.19 Sulfuric Acid 18N: Slowly add 500 mL of concentrated H2SO4 (stored under hood in glassware room) to 500 mL of reagent water in pyrex 2L beaker while under a hood.
CAUTION: This solution will get very hot. Do not attempt to do this in plastic beakers because they will melt. Only a large Pyrex

beaker should be used. It will take time to bring it down to ambient temperature for use in the test.

7.2.20 Calcium hypochlorite solution (0.35M) $Ca(OCl)_2$: Combine 5 g of calcium hypochlorite (Stored on reagent shelf in wet lab) and approximately 50 mL of water in a 100mL volumetric flask. Bring to volume with DI water Shake before using. Store under hood in wet lab.

7.2.21 Sodium hydroxide solution (1.25N) NaOH: Dissolve 50 g of NaOH (stored under hood in wet lab)into DI water in a 1L volumetric flask. Bring to volume with DI water. Store under hood in wet lab.

7.2.22 Hexane: ACS grade (Stored at ambient temperature in extraction lab).

7.2.23 Sodium hydroxide solution (50%) NaOH: Dissolved 50g of NaOH (stored under hood in wet lab) in 100mL of DI water in a large pyrex beaker. **CAUTION: This solution will get very hot.** Do not attempt to do this in plastic beakers because they will melt. Only a large Pyrex beaker should be used. It will take time to bring it down to ambient temperature for use in the test. Store under hood in wet lab.

7.2.24 Acetic Acid (1.6M): Into a 1L volumetric flask, combine 92mL of glacial acetic acid (stored at ambient temperature under the counter in the extraction lab) with 500mL of DI water. Bring to volume with DI water.

8.0 INSTRUMENT OR METHOD CALIBRATION

See Sample preparation and analysis section 10.0.

9.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

9.1 A minimum of 50 mL of sample is required for liquid analysis. Samples must be preserved with sodium hydroxide to pH of >12 , and cooled to 4 ^{o} C at the time of collection (approximately 2 mL NaOH/1-L sample). Soil samples can be taken in unpreserved 4 oz. jars and cooled to 4º C until analysis.

9.2 If sample contains chlorine or hydrogen sulfide, see section 6.0 for treatment. Document any sample pretreatment in sample prep book.

9.3 Samples may not be stored for longer than 14 days.

10.0 SAMPLE PREPARATION AND ANALYSIS

10.1 Pretreatment for Cyanides Amenable to Chlorination

10.1.1 To one 50-mL sample or to a sample diluted to 50 mL, add calcium hypochlorite solution dropwise while stirring. Maintain the pH of the sample between 11 and 12 with 1.25N NaOH. Test for chlorine with KI starch paper by adding a drop of sample to a test strip. When excess chlorine is present a drop of sample added to the paper will turn it blue. **CAUTION: Cyanogen chloride**

may form during this reaction. This is a very toxic gas and this step should always be performed in a hood.

10.1.2 Maintain this excess chlorine for one hour with continuous stirring. Periodically spot check with the KI paper and add more calcium hypochlorite solution as necessary.

10.1.3 After one hour, add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.03 g of ascorbic acid to each 50 mLs of sample. Note this in the sample prep book.

10.1.4 Test for total cyanide as described in the following procedure in both the chlorinated and the unchlorinated samples. The difference of total cyanide in the chlorinated (listed as Cyanide + Chlorine in LIMS) and the unchlorinated (listed as Cyanide or Total Cyanide in LIMS) samples is the cyanide amenable to chlorination.

10.2 Distillation Procedure:

10.2.1 Pour 50 mL 0.25 M NaOH into each scrubber tube.

10.2.2 For liquid samples, add 50 mL of sample or an aliquot diluted to 50 mL to the midi-distillation boiling tube. For soil samples: weigh up approximately 2 g of soil and place in boiling tube, then add 50 mLs of 0.25 NaOH. Add PTFE boiling chips as required.

10.2.3 Add 0.2 g of sulfamic acid to tube before assembly of apparatus.

10.2.4 Low Distillation Standard (LCG / QSM requirement): add 50 ml of 30ug/L working standard to midi-distillation boiling tube.

10.2.5 High Distillation Standard (LCG / QSM requirement): add 50 ml of 100ug/L working standard to midi-distillation boiling tube.

10.2.6 Assemble each apparatus. Start the vacuum and water after all connections are made and checked. Adjust the vacuum bubble rate at approximately 2 bubbles/ second. If you are distilling a soil the bubble rate should be higher to ensure adequate mixing of reagent with the soil in suspension. Let bubbles mix the solution for about 3 minutes. For spiked samples, add 0.25 mL of 10mg/L cyanide standard with a calibrated pipettor through the air inlet at this point. Rinse down with a small amount of reagent water, then let the solution mix for 3 minutes.

10.2.7 Slowly add 5 mL of 18N H_2SO_4 through the air inlet tube. Rinse down with a small amount of reagent water, continue to bubble 3 minutes. Add 2 mL of Magnesium Chloride (6.7) into the inlet tube and wash down with a small amount of reagent water.

10.2.8 Turn on the Easy-Dist. Set the controller to the following settings: Rate1: LO, Temp1: 190^0C, Time1: 2.5 hours, Time2: 0.

10.2.9 Press Start. The system should be monitored periodically to make sure the apparatus is working correctly.

10.2.10 After distillation time finishes, continue the vacuum and water flow until the temperature displayed for the Easy-Dist is less than 100 ^{0}C.

10.2.11 Disconnect the absorber tube, shut off the vacuum and remove the absorber tube.

10.2.12 Pour off the trapped cyanide solutions into 50 mL twistcap plastic centrifuge tubes. Prepared samples can be stored in a tightly lidded container, refrigerated at 4°C in the walk-in cooler until analysis.

10.3 Calibration and Analysis:

10.3.1 Set up Lachat programs to include the following timing guidelines. Save as the appropriate method. Load program needed for analysis.
Injection Timing:
Pump Speed: 35
Cycle Period: 60 sec.
Sample loop: 150 cm
Load Period: 30 sec.
Inject period: 30 sec.
Inject to start of peak: 10 sec.

10.3.2 Turn on and set heating unit at 60°C. It should take approximately 15 minutes to reach desired temperature.

10.3.3 Inspect modules for proper connections.

10.3.4 Turn on power to all modules.

10.3.5 Place reagent lines into proper containers. Apply tension on pump tube cassettes. Refer to Lachat Manuals for specific details and a diagram of the manifold.

10.3.6 Pump system until a stable baseline is attained.

10.3.7 Place calibration standards and blank in sample tray in descending order of concentration followed by check standards and unknowns. The concentrations of the unknowns (samples) as well as the check standards and other QC will be evaluated in relation to the absorbencies of the calibration standards. The Lachat program prints out the raw data in ug/L.

10.3.8 For samples that have results higher than the highest calibration standard, dilute samples using 0.25M NaOH as the diluent and reanalyze.

10.3.9 At end of run, place all transmission lines in water, and flush system. Pump lines dry.

10.3.10 Turn off pump, all modules, and release tension levers on pump tube cassettes.

10.3.11 Clean area thoroughly.

11.0 TROUBLESHOOTING AND MAINTENANCE

Refer to Lachat and Easy-Dist operation manuals for troubleshooting and maintenance instructions.

12.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

Liquid Concentration (ug/L) = A x C

$$\textit{Solid Concentrations (mg/kg)} = \frac{A \times B \times C}{D \times E},$$

where A = instrument reading for sample (ug/L)
B = total volume of digestion (L)
C= analyst dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary

$$\textit{Spike Recovery (\%)} = \frac{\text{(Spiked sample concentration – Sample concentration)} \times 100}{\text{(Spike amount)}}$$

$$\textit{\%RPD} = \frac{(MS - MSD) \times 100}{(MS + MSD)/2}$$

where MS = Matrix spike concentration
MSD = Matrix spike duplicate concentration

13.0 COMPUTER HARDWARE AND SOFTWARE

13.1 Computer with StarLIMS
13.2 Lachat QuickChem 8000 software

14.0 DATA MANAGEMENT AND RECORD MANAGEMENT

14.1 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.
14.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
14.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.
14.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

15.0 QUALITY CONTROL AND QUALITY ASSURANCE

15.1 The analyst must show an initial demonstration of capability (IDC) to generated acceptable data, by successfully analyzing four replicates of a known standard and having acceptable results for a blind sample.
15.2 ICV (Initial Calibration Verification): This mid range check standard is analyzed after calibration. Control limits are +/- 10% of the true value (+/- 15 % for QSM). If the recovery is outside of this range, terminate the run and correct the problem before proceeding.
15.3 ICB (Initial Calibration Blank): Analyzed immediately after the ICV. The value of the ICB must be below the method LOD. If not, terminate the analysis and correct the problem before proceeding or qualify results less than 20 times the ICB with a 'B' flag.
15.4 LCS (Laboratory Control Sample): Analyze a LCS (reference obtained from a second source) per 20 samples. Control limits are +/- 15% of the

true value. An out of control LCS may indicate that the calibration standard stock solution is inaccurate or that there was a problem with the distillation process. If the recovery exceeds this, terminate the analysis and correct the problem before proceeding

15.5 CCV (Continuing Calibration Verification): Analyze a CCV following every ten samples and at the end of the analysis. Control limits are +/- 10% of the true value (+/- 15 % for QSM). If recovery is outside these limits, recalibrate and reanalyze all samples back to the last acceptable CCV or ICV.

15.6 CCB (Continuing Calibration Blank): Analyzed immediately after the CCV. The value of the CCB must be below the method LOD. If not, terminate the analysis and correct the problem before proceeding. If CCB is outside these limits, recalibrate and reanalyze all samples back to the last acceptable CCB or ICB or qualify results less than 20 times the CCB with a 'B' flag.

15.7 MS-MSD (Matrix Spike-Matrix Spike Duplicate): A MS-MSD is required every analytical run at a frequency of 5% (1 for every 20 samples) per matrix type. For QSM work an MS-MSD is required with every ten samples analyzed. Refer to the control limits for acceptance criteria for both accuracy and precision. Unless otherwise specified in a contract if recovery is outside of acceptable range, flag the sample with an 'M' qualifer. If the RPD is outside of the acceptable range, flag the sample with a 'Y' qualifier.

15.8 Duplicate: One sample per batch of 20 samples is run in duplicate. The RPD between the sample and its duplicate must be within in-house or project/program specific limits or data is qualified (Y). The limit for QSM work is 20 % RPD.

15.9 MB (Method Blank): analyze one method blank per digestion batch. This is a reagent water blank (or Sand w/ reagent water) that is taken through all of the processes that the samples are taken. The value of the method blank must be below the method LOD/RL , or the program/project specified limits. The MB must be < ½ the RL for QSM work. If not, terminate the analysis and correct the problem before proceeding or qualify results less than 20 times the method blank with a 'B' flag.

15.10 Low Distillation Standard(LCG / QSM requirement): prepare once per calibration. Recovery should fall within +/-10 % for LCG data and +/- 15 % for QSM data. An out of control standard may indicate there was a problem with the distillation process. If the recovery exceeds these limits then terminate the analysis and correct the problem before proceeding with repreparation and analysis.

15.11 High Distillation standard (LCG / QSM requirement): prepare once per calibration. Recovery should fall within +/-10 % for LCG data and +/- 15 % for QSM data. An out of control standard may indicate there was a problem with the distillation process. If the recovery exceeds these limits then terminate the analysis and correct the problem before proceeding with repreparation and analysis.

15.12 1000 mg/L CN stock standard is standardized against a 0.0192N $AgNO_3$ solution biweekly. Acceptance criteria, is 995 – 1005 mg/L CN.

15.13 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

16.0 REFERENCES

16.1 *SW 846*, 3rd Edition, Update III, 1997 Method 9010B, 9012A and 9013.

16.2 *EASY-Dist Environmental Methods Manual*, Rev 6/23/97, Cyanide Methods

16.3 *Lachat QuickChemAE Operating Manual*, Issued: 10/01/93

16.4 *Lachat QuickChem Methods Manual*, Revision Date: 18 January 1996

16.5 *Lachat QuickChemAE Software Reference Manual*, 24 January 1994

16.6 *Department of Defense, Quality Systems Manual for Environmental Laboratories, DoD Environmental Data Quality Workgroup, Department of Navy, Lead Service, Draft Version 3, December 2004*

16.7 *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA-600/R-93/ 100, August 1993, Method 335.4.

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Six-point initial calibration plus blank (ICAL)	Initially and as needed	r $\geq$ 0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis (Second Source Std.)	%R: 85-115% of true value, or program / project specific, or 90-110 %R for Drinking water	Remake and reanalyze ICV standard, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	If required, after each ICV, prior to sample analysis	< Reporting Limit (RL)	Remake and reanalyze , if still unacceptable investigate and correct problem.
Continuing calibration verification (CCV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R: 85-115% of true value, or program / project specific, or 90-110 %R for Drinking water	Remake and reanalyze CCV, if still unacceptable investigate and correct problem. Reanalyze all samples after last acceptable CCV.
Method Blank (MB)	One (1) per analytical batch	<MDL or <20 times less than the lowest sample result, or program / project specific, or LCG / QSM: <1/2 RL	Clean analytical system, repeat. If still unacceptable, redistill batch or flag all results less than 20 X's the MB with a 'B' qualifier.
Sample duplicate (QSM requirement)	One (1) per analytical batch (20 Samples) per matrix	<20 % RPD	Investigate problem, if none apperant qualify results. Qualify results with a 'Y' flag if necessary
Matrix Spike (MS) – Matrix Spike Duplicate (MSD)	One (1) each per analytical batch per matrix DOD-QSM: One (1) each per ten samples	In-house, program-project specified limits, or QSM: LCS recovery criteria, RPD < 20 %.	Investigate problem, if LCS in control qualify results. Qualify results with a 'M' and/or 'Y' flag if necessary
Low Distillation Standard (LCG / QSM requirement)	One (1) per calibration	Within +/- 15 % of true value	Troubleshoot and reanalyze. If fails again redistill samples
High Distillation Standard (LCG / QSM requirement)	One (1) per calibration	Within +/- 15 % of true value	Troubleshoot and reanalyze. If fails again redistill samples
Laboratory control sample (LCS)	One per analytical batch	In-house, program, or project specific limits (where applicable)	Halt analysis, troubleshoot and reanalyze. If still unacceptable, redistill batch

Data Validation Checklist

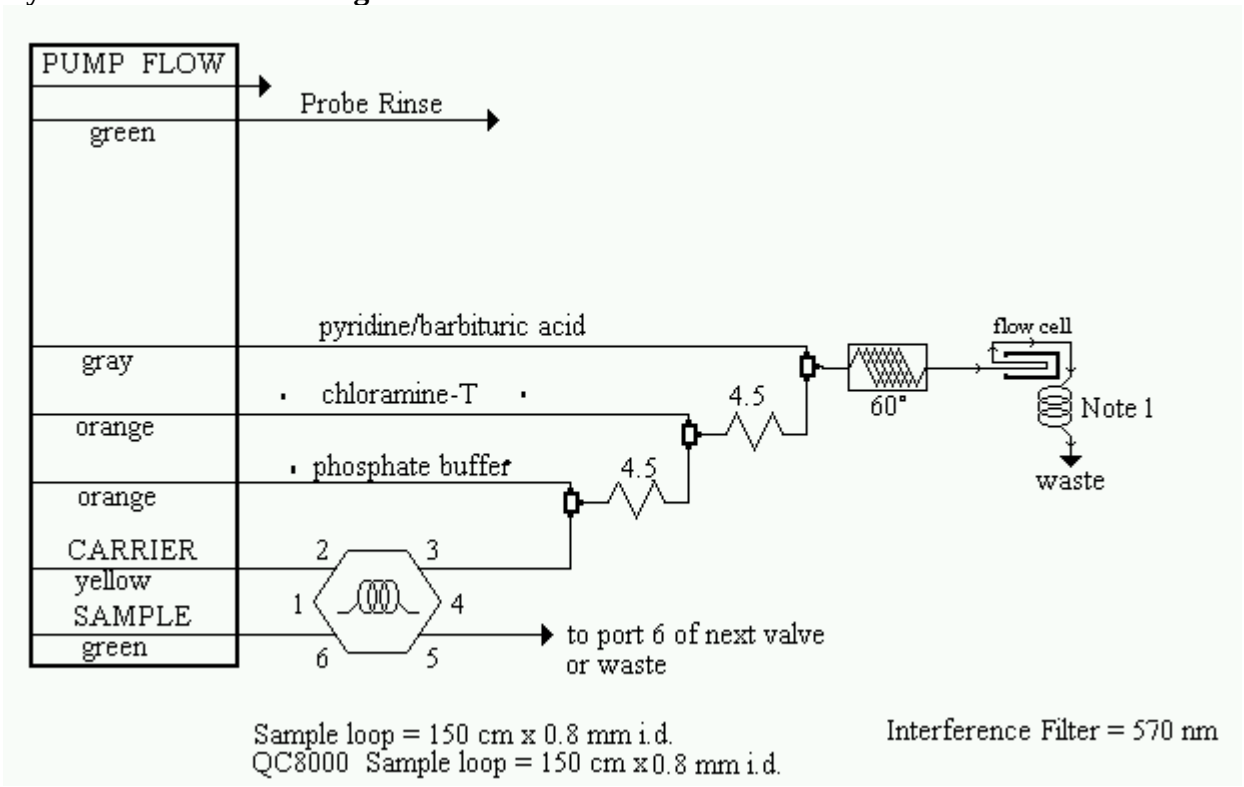
LIMS #:	Method: Cyanide			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

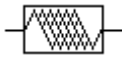
Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a distillation sheet accompany the data package?	---					
2. Does the distillation sheet include all reagent and standard prep log numbers?	---					
3. Is a copy of the stock standard standardization log included?	---					
4. Were samples checked for chlorine and sulfide?	---					
5. Were the samples distilled within hold time?	14 days					
6. Was a method blank prepared at the required frequency?	1per 20 samples					
7. Was the method blank result acceptable?	< LOD					
8. Was a LCS prepared at the required frequency?	1per 20 samples					
9. Was the LCS used before the indicated expiration date?	---					
10. Was the LCS recovery acceptable?	Within in house, project, or LCG/ QSM QC limits					
11 Was the MS and MSD prepared at the required frequency?	1per 20 samples (1 per 10 for QSM) per matrix					

Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
12. Were the MS and MSD recoveries acceptable?	Within in house, project, or QSM QC limits					
13. Was the RPD between the MS and MSD acceptable?	Within in house QC limits					
14. Was calibration performed using required number of standards	6 plus a blank					
15. Is the standard prep log number noted on the analytical report?	---					
16. Was the correlation coefficient (r) acceptable?	> or = 0.995					
17. Were distilled High/Low standard recoveries acceptable (QSM/LCG)?	85 – 115 % Rec.					
18. Were the ICV and ICB (if required) run immediately after the calibration curve?	---					
19. Was the ICV recovery acceptable?	85-115 % rec. (90-110 % for Drinking water)					
20. Was the ICB result acceptable?	< MDL / RL					
21. Were the CCV's analyzed at the required frequency?	1per 10 samples					
22. Were the CCV recoveries acceptable?	85-115 % rec. (90-110 % for Drinking water)					
23. Was a MB analyzed at the required frequency?	1 per 20 per matrix					
24. Was the MB results acceptable?	< MDL / RL, project specific, or <1/2 RL for LCG / QSM					
25. Was a sample run in Duplicate and was precision acceptable (QSM/LCG data)?	+/- 20 % Diff.					
26. Are all samples on the job lists accounted for?	---					

Cyanide MANIFOLD diagram



CARRIER is 0.25 M sodium hydroxide solution (Reagent 1).
All manifold tubing is **0.8 mm (0.030 in) i.d**. This is **5.2 uL/cm**.
4.5 is **70** cm of tubing on a **4.5** cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module are required. The  shows **650 cm of tubing** wrapped around the heater block at the specified temperature.
Note 1: 2 meter back pressure loop, 0.52 mm i.d.

CT Laboratories
Baraboo Laboratory Division

Title: **Semi-Volatile Explosives by Method 8330 with extended analyte list**

SOP Number: **8330B**

Written by: _____

Technical Review by: _____

Approved by: _____
Quality Assurance Manager

Approved by: _____
Laboratory Director

SOP Manual Control Number: _____

1.0 SCOPE/APPLICATION

1.1 Method SW-846 8330B-extended analyte list is used to determine the quantitation of various explosive compounds in extracts from solid and aqueous matrices. High Performance Liquid Chromatography (HPLC) is employed with two columns and a dual wavelength UV detector. Target compounds determined by this method are listed in Table 1.0 with current method detection limits.

1.2 This method is a dual column analysis. Each column uses the same QC criteria for the reporting of analytical results. The analytical columns are joined to a single injection port. The secondary chromatographic column is used to confirm or report final results in conjunction with the primary column.

1.3 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

1.4 This method is used to determine the concentration of certain Nitroaromatics and Nitroamines by High Performance Liquid Chromatography (HPLC) in water and soil matrices. Specifically, the following compounds:

Table 1.0
Explosives with Method Detection Limits and Limits of Quantitation

Analytes	Water MDLs (ug/L)			Soil MDLs (ug/kg)	
	MDLs	LOQs		MDLs	LOQs
HMX	0.26	0.87		63	208
RDX	0.21	0.70		52	172
1,3,5 DNB	0.24	0.80		61	201
1,3 DNB	0.25	0.82		37	122
Tetryl	0.18	0.61		69	229
Nitrobenzene	0.25	0.83		43	141
2,4,6 TNT	0.28	0.93		72	238
4-Am-DNT	0.27	0.89		73	243
2-Am-DNT	0.23	0.78		53	176
2,6-DNT	0.25	0.83		58	194
2,4-DNT	0.30	0.99		39	130
2-NT	0.28	0.93		41	136
3-NT	0.28	0.92		64	211
4-NT	0.27	0.89		69	229
Nitroglycerin (NG)	2.3	7.7		581	1936
PETN	4.4	14.5		775	2583
Nitroguanidine (NQ)	26	84		368	1225

Table 1.1
Explosives and Retention Times

Explosives Name	Primary Column Retention Time (min.)	Secondary Column Retention Time (min.)
HMX	5.391	5.003
RDX	8.716	6.431
1,3,5 DNB	11.358	15.111
1,3 DNB	14.791	11.651
Tetryl	15.607	23.424
Nitrobenzene	17.074	8.464
2,4,6 TNT	18.549	24.522
4-Am-DNT	20.557	13.241
2-Am-DNT	21.507	14.629
2,6-DNT	22.899	16.339
2,4-DNT	24.032	17.229
2-NT	26.948	12.251
3-NT	29.831	13.684
4-NT	28.607	12.598
1,2-DNB (SURR)	12.458	8.986
NG	20.36	10.946
PETN	32.68	27.022
NQ	3.903	2.250

Note: Retention time shifts can occur when instrument maintenance is performed. The shifts in the retention times are reflected in the analytical method.

Table 1.2
LCS Control Limits

	LCG					QSM			
	Water (ug/L)		Soil (mg/kg)			Water (ug/L)		Soil (mg/kg)	
Analyte	Lower Limit	Upper Limit	Lower Limit	Upper Limit		Lower Limit	Upper Limit	Lower Limit	Upper Limit
HMX	45	140	60	140		80	115	75	125
RDX	39	120	59	120		50	160	70	135
1,3,5 DNB	53	135	50	135		65	140	75	125
1,3 DNB	54	120	50	120		45	160	80	125
Tetryl	30	120	37	120		20	175	10	150
Nitrobenzene	49	120	50	120		45	160	80	125
2,4,6 TNT	37	120	44	120		50	145	55	140
4-Am-DNT	58	159	50	159		55	155	80	125
2-Am-DNT	53	120	50	120		50	155	80	125
2,6-DNT	52	144	50	144		60	135	80	120
2,4-DNT	58	136	50	136		60	135	80	125
2-NT	52	120	59	120		45	135	80	125
3-NT	48	136	50	136		50	130	75	120
4-NT	46	136	58	136		50	130	75	125
NG	50	150	50	150		50	150	50	150
PETN	50	150	50	150		50	150	50	150
NQ	50	150	50	150		50	150	50	150

Table 1.3
Matrix Spike/Matrix Spike Duplicate Limits

	LCG						QSM					
	Water (ug/L)			Soil (mg/kg)			Water (ug/L)			Soil (mg/kg)		
Analyte	**Lower Limit**	**Upper Limit**	**RPD**	**Lower Limit**	**Upper Limit**	**RPD**	**Lower Limit**	**Upper Limit**	**RPD**	**Lower Limit**	**Upper Limit**	**RPD**
HMX	45	140	30	60	140	50	80	115	30	75	125	30
RDX	39	120	30	59	120	50	50	160	30	70	135	30
1,3,5 DNB	53	135	30	50	135	50	65	140	30	75	125	30
1,3 DNB	54	120	30	50	120	50	45	160	30	80	125	30
Tetryl	30	120	30	37	120	50	20	175	30	10	150	30
Nitrobenzene	49	120	30	50	120	50	50	140	30	75	125	30
2,4,6 TNT	37	120	30	44	120	50	50	145	30	55	140	30
4-Am-DNT	58	159	30	50	159	50	55	155	30	80	125	30
2-Am-DNT	53	120	30	50	120	50	50	155	30	80	125	30
2,6-DNT	52	144	30	50	144	50	60	135	30	80	120	30
2,4-DNT	58	136	30	50	136	50	60	135	30	80	125	30
2-NT	52	120	30	59	120	50	45	135	30	80	125	30
3-NT	48	136	30	50	136	50	50	130	30	75	120	30
4-NT	46	136	30	58	136	50	50	130	30	75	125	30
NG	50	150	30	50	150	50	50	150	30	50	150	50
PETN	50	150	30	50	150	50	50	150	30	50	150	50
NQ	50	150	30	50	150	50	50	150	30	50	150	50

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for isolating Nitroaromatic and Nitroamine compounds through sample preparation from aqueous and solid matrices (reference methods SW846-3535, 8330B soil extraction, and USACCREL nitroguanidine sample preparation).

2.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. The resulting solution is filtered and analyzed on HPLC that is equipped with a dual wavelength UV detector and employs a dual column technique.

2.3 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

3.0 DEFINITIONS

3.1 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process

3.2 Laboratory Control Spike (LCS): Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedures. The laboratory control spike is used to document the ability of an analyst to generate acceptable precision and bias to verify the analytical system performance, and to document method accuracy for each matrix.

3.3 Laboratory Control Spike Duplicate (LCSD): A duplicate Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The laboratory control spike duplicate is used to document the precision and bias of a method. This default QC type is only performed when insufficient amount of sample is available for performing a matrix spike and matrix spike duplicate.

3.4 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. Matrix spikes are used to document the precision and bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): Intra-laboratory split samples spiked with identical concentrations of target analytes. The spiking occurs prior to sample preparation and analysis. Matrix spike duplicates are used to document the precision and bias of a method in a given sample matrix.

3.6 Method Reporting Limit (MRL): The method reporting limit is a threshold value below which the laboratory reports a result as non-detect. The highest value reported for the method reporting limit is dependant upon project-specific action or decision levels. Method reporting limits are adjusted based on the sample matrix and any sample dilution/concentrations when necessary.

3.7 Method Detection Limit (MDL): The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. It is determined from analysis of a sample in a given matrix containing the analyte. Depending on project specifications, MDLs will be in form of a MDL check standard or the MDL is subject to extraction procedures. An acceptable MDL standard check must produce a signal at least 3 times the instrument's noise level. Also, a MDL check injection must be made after each major instrument repair or changing of instruments (prior to sample analysis) to determine the performance sensitivity of the analysis.

3.8 Surrogate (SURR) An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. The surrogate is used to monitor the performance of the extraction, cleanup (as needed), the analytical system, and the effectiveness of the method. The surrogate is 1,2-Dinitrobenzene.

3.9 Initial Calibration (ICAL): An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of an analyte. This relationship is depicted graphically, and referred to as a "calibration curve". Initial calibration curves must be established based upon the requisite number of standards

identified within the method for each target analyte.

3.10 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed.

3.11 Continuing Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems can be necessary. Refer to each method for specific guidance on quality control procedures.

4.2 4-Am-DNT and 2-Am-DNT elute at similar retention times. Therefore, a large concentration of one isomer will mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture shall be reported.

4.3 Tetryl is an unstable compound that decomposes rapidly, especially in water/methanol solutions and with heat. In addition, the degradation products of tetryl can appear as a shoulder on the peaks of other compounds. Water samples suspected of containing tetryl can be diluted with acetonitrile and acidified to pH<3 to help stabilize the sample. Also, samples containing tetryl will not be exposed to temperatures above room temperature.

5.0 SAFETY

5.1 Gloves and protective clothing will be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure will utilize appropriate laboratory safety systems.

5.2 The toxicity of chemicals used in this method has not been precisely defined. Each chemical will be treated as a potential health hazard, and exposure to these chemicals will be minimized.

5.3 Because of the explosive nature of some of the target analytes and their derivatives, special care will be taken when handling standard neat materials and in the infrequent cases where water and soil samples are highly contaminated. Do not dry standards and samples at oven temperatures, and do not grind with mortar and pestle suspected highly

contaminated soils. It is recommended that soil and waste samples suspected as being highly contaminated be screened first using EPA Method 8515. Visual inspection of solid samples is very important and any samples having a chemical appearance or appear as a finely ground grayish-white material shall be treated as contaminated.

6.0 APPARATUS & MATERIALS

Apparatus:

6.1 High Performance Liquid Chromatograph (Shimadzu)- An analytical system complete with auto-injection and all required accessories including syringes, analytical columns, dual wavelength UV detector, degasser, and EZ Chrom Elite data acquiring system.

6.2 Primary column: Restek Pinnacle II C18, 5 um, 250x4.6 mm, catalog #9214575.

6.2.1. HPLC parameters:

Mobile phase = 54% MeOH/46% H2O
Run time = 35 minutes
Injection Volume: 100 uL
Flow rate = 1.0 mL/min
Wavelength = 254 nm and 210 nm (excluding nitroguanidine)
Nitroguanidine wavelength = 263 nm and 254 nm

6.3 Secondary column: Phenomenex Luna Phenyl-Hexyl, 5 um, 250x4.6 mm, part number 00G4257-E0.

6.3.1. HPLC parameters:

Mobile phase = 61.0% MeOH/39.0% H2O
Run time = 35.5 minutes
Injection Volume: 100 ul.
Flow rate = 1.4 mL/min
Wavelength = 254 nm and 210 nm (excluding nitroguanidine)
Nitroguanidine wavelength = 263 nm and 254 nm

6.4 Pre-columns: Restek Pinnacle II C18, 5 um, 20x4.0 mm, catalog #921450220 or equivalent for primary column. Restek Pinnacle DB Phenyl, 5 um, 20x4.0 mm, catalog #941550220 or equivalent for secondary column.

Note: Instrument operating parameters and mobile phase composition are subject to change to improve overall chromatography (note changes in the Instrument Maintenance Logbook).

6.5 Solid Phase Extraction (SPE) Manifold (Visiprep, Supelco) -2000 mL side arm flask. -10-25 mL test tubes (glass/ptfe).

6.6 SPE extraction cartridges (BondElut, PPL, #1225-5001, 6 mL, 500 mg or equivalent).

6.7 Syringes (disposable) B-D #301604, 10-mL plastic syringe or equivalent.

6.8 Filtration discs (disposable) Whatman 0.45micron (#6788-1304), 13 mm or equivalent.

6.9 Analytical balances (Fisher, #XD-2200) -capable of accurately weighing to 0.0001 grams (for standard preparation) and to the nearest 0.01 gram (for sample preparation).

6.10 Spatulas- stainless steel.

6.11 Vortex mixer (VWR #g-560).

6.12 Teflon sampler (Supelco #59410)-used in conjunction with SPE manifold for adding sample to SPE cartridges.

6.13 Flow control liners (Supelco # 57059)-disposable-TFE.

6.14 Mortar and pestle (Fisher #12990).

6.15 Ultrasonic bath (Fisher, #FS-28)-temperature controlled (<30 C) by placing a coil of copper tubing, connected to the well or tap water faucet, into the bath water and circulating cold tap water through the coil during the entire sonication process.

Glassware:

6.16 Graduated cylinders – 100 mL, 500 mL, & 1000 mL (Pyrex).

6.17 Beakers - 150 mL, 250 mL, 600 mL (Pyrex, Kimax).

6.18 Vials - 2 mL (NSC, # 4000-186w) and 15 mL (Supelco, #27003) amber screw cap vials with Teflon lined caps, 20 mL (Fisher, #033374) vials (glass), 60 mL amber jars with Teflon lined caps.

6.19 Pasteur Pipettes (VWR, #53283-911).

6.20 Syringes (glass) 10 uL (Hamilton #24557), 100 uL (#24563), and 1.0 mL (#24575) or equivalent.

6.21 Volumetric flask - Class A 10, 25, 50, and 100 mL (Pyrex, Kimax).

6.22 Vortex tubes (Pyrex, # 264828)-15 mL, glass or equivalent.

Reagents:

6.23 Deionized water (In-house MilliQ processed reagent water).

6.24 Silica sand- analyte free. Purify by heating at 400° C for 4 hours in a shallow glass

tray.

6.25 Calcium Chloride, 4-20 mesh, anhydrous (Fisher C614-500), or equivalent

6.26 Acetonitrile (Mallinckrodt #2856-10), HPLC grade

6.27 Methanol (Fisher #A452-4), HPLC grade

7.0 STANDARDS AND SPIKES

7.1 Preparation of standards is documented in the Explosives standards logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards, once opened, expire within six months or sooner if routine QC indicates a problem and are not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards, which are subsequent dilutions made from the opened stock standard vial, expire within six months and are not to exceed the opened date of the stock standard or the manufactures expiration date.

7.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standards shall have the manufactures suggested expiration date. Stock standards, once opened, expire within six months and are not to exceed the manufactures expiration date. The following list of stock standards are commercially prepared certified standards, such as:

8330 Calibration Mix 1: Restek #31450 at 1000 ug/ml
8330 Calibration Mix 2: Restek #31451 at 1000 ug/ml
8330 Surrogate: Restek #31453 at 1000 ug/ml
PETN standard: Restek #31600 at 1000 ug/ml
Nitroglycerin standard: Restek #31498 at 1000 ug/ml

Since nitroguanidine powder contains a certain percentage of water, this must be accounted for when making the stock solutions. The exact water percentage may be obtained from the certificate of analysis that is received with each new bottle of nitroguanidine that is purchased. Nitroguanidine stock solution is prepared at a final concentration of 1000 ug/ml in Millipore water. Nitroguanidine is purchased as a solid material from which stock standard solutions must be prepared for both the primary and secondary sources:

Nitroguanidine solid source 1: Aldrich #N17351
Nitroguanidine solid source 2: Chem Service #6380

7.3 Intermediate Working Stock Standards: These standards are diluted stock standards so that the concentration levels are manageable for the working range of the HPLC. The explosives standards are prepared at an optimum level from the stock standard. Each explosive compound from Mix 1 and Mix 2, including the surrogate is at a concentration of 10.0 ug/ml (Table 3.0). Nitroglycerin, PETN, and nitroguanidine solutions are also

prepared at a concentration of 10.0 ug/ml.(Table 3.1) and nitroglycerin and PETN at 100.0 ug/ml.

Table 3.0
Explosives Mix 1 & 2 Intermediate Stock Standard Concentration

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Cal Mix 1	1000	0.10	10	10.0
Cal Mix 2	1000	0.10	10	10.0
Surrogate	1000	0.10	10	10.0

Note: The concentration of the Intermediate Working standard and Intermediate ICV standard are prepared in the same manner.

Table 3.1
NG, PETN, and NQ Intermediate Stock Stadards

Intermediate Standards	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Diluting Solvent	Final Volume (ml)	Final Concentration (ug/ml)
Nitroglycerin (NG) low level	1000	0.10	ACN	10	10.0
Nitroglycerin (NG) high level	1000	1.000	ACN	10	100.0
PETN low level	1000	0.10	ACN	10	10.0
PETN high level	1000	1.000	ACN	10	100.0
Nitroguanidine (NQ) low level	1000	0.10	Millipore H2O	10	10.0
Nitroguanidine (NQ) high level	1000	1.000	Millipore H2O	10	100.0

7.4 Calibration standards: An initial calibration of the listed explosive compounds in Table 1.0 is performed using a minimum of 5 calibration points. Such standards will be used to demonstrate the linearity of the UV detector corresponding to the expected concentrations found in real samples. The calibration standards are made by taking known aliquots of the intermediate standard and diluting them to volume in a 1:1 solution of acetonitrile: 5g/L Calcium Chloride solution. They are stored in the dark and refrigerated and must be used within one day of preparation. Alternative standard concentrations for the calibration curve can be prepared to meet client or program specified criteria.

Linearity points for Mix 1 & 2 Explosives

Level 1	0.02 ug/ml
Level 2	0.05 ug/ml
Level 3	0.10 ug/ml
Level 4	0.50 ug/ml
Level 5	1.00 ug/ml
Level 6	2.00 ug/ml
Level 7	2.50 ug/ml

Linearity points for Nitroguanidine

Level 1	0.05 ug/ml
Level 2	0.10 ug/ml
Level 3	0.50 ug/ml
Level 4	1.00 ug/ml
Level 5	2.00 ug/ml
Level 6	2.50 ug/ml

Linearity points for Nitroglycerin and PETN

Level 1	0.20 ug/ml
Level 2	0.50 ug/ml
Level 3	1.00 ug/ml
Level 4	2.00 ug/ml
Level 5	5.00 ug/ml
Level 6	10.00 ug/ml
Level 7	15.00 ug/ml

Table 3.2
Calibration Points for Mix 1 & 2 Explosives Linearity

Linearity Points	Spike Concentration (ug/ml) Intermediate	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
1	10.0	0.002	1.0	0.02
2	10.0	0.005	1.0	0.05
3	10.0	0.010	1.0	0.10
4	10.0	0.050	1.0	0.50
5	10.0	0.100	1.0	1.00
6	10.0	0.200	1.0	2.00
7	10.0	0.250	1.0	2.50

Table 3.3
Calibration Points for Nitroguanidine

Linearity Points	Spike Concentration (ug/ml) Intermediate	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
1	10.0	0.005	1.0	0.05
2	10.0	0.010	1.0	0.10
3	10.0	0.050	1.0	0.50
4	10.0	0.100	1.0	1.00
5	10.0	0.200	1.0	2.00
6	10.0	0.250	1.0	2.50

Table 3.4
Calibration Points for Nitroglycerin and PETN Linearity

Linearity Points	Spike Concentration (ug/ml) Intermediate	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
1 NG	10.0	0.025	1.0	0.25
PETN	10.0	0.035	1.0	0.35
2 NG	10.0	0.050	1.0	0.50
PETN	10.0	0.070	1.0	0.70
3 NG	10.0	0.100	1.0	1.00
PETN	10.0	0.100	1.0	1.00
4 NG	10.0	0.200	1.0	2.00
PETN	10.0	0.200	1.0	2.00
5 NG	100.0	0.050	1.0	5.00
PETN	100.0	0.050	1.0	5.00
6 NG	100.0	0.100	1.0	10.00
PETN	100.0	0.100	1.0	10.00
7 NG	100.0	0.150	1.0	15.00
PETN	100.0	0.150	1.0	15.00

7.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The ICV standard involves the analysis of all the target compounds each time the initial calibration is performed. The ICV is prepared the same way and at the same concentration as the calibration point 4 of the calibration curve.

Table 3.5
Mix 1 & 2 Explosives ICV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Mix 1 & 2 Intermediate	10.0	0.05	1.0	0.50

Table 3.6
Nitroguanidine ICV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
NQ	10.0	0.05	1.0	0.50
Surrogate	10.0	0.05	1.0	0.50

Table 3.7
Nitroglycerin and PETN ICV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
NG	10.0	0.20	1.0	2.00
PETN	10.0	0.20	1.0	2.00
Surrogate	10.0	0.05	1.0	0.50

7.6 Continuing Calibration Verification (CCV) Standard: A working standard solution used to check the validity of a calibration curve on a daily basis. The CCV is prepared the same way and at the same concentration as the calibration point 4 of the calibration curve.

Table 3.8
Exploisve Mix 1 & 2 CCV Working Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Mix 1 & 2 Intermediate	10.0	0.25	5.0	0.50

Table 3.9
Nitroguanidine CCV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
NQ	10.0	0.25	5.0	0.50
Surrogate	10.0	0.25	5.0	0.50

Table 3.10
Nitroglycerin and PETN CCV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
NG	10.0	1.00	5.0	2.00
PETN	10.0	1.00	5.0	2.00
Surrogate	10.0	0.25	5.0	0.50

7.7 Spiking standard: The explosives mix spiking standard is prepared by diluting the 8330 calibration mix 1 and 2 in acetonitrile to produce a working spiking standard of 10.0 ug/mL. 0.5 mL is added to the quality control and matrix spike water samples and 1.0 mL is added to the quality control and matrix spike soil samples. For nitroguanidine, the intermediate standards (table 3.1) are used as spiking standards. 0.500 ml of the 10.0 ug/ml low level NQ intermediate standard is added to the quality control and matrix spike water samples. 0.500 ml of the 100.0 ug/ml high level NQ intermediate standard is added to the quality control and matrix spike soil samples. The nitroglycerin and PETN high level (100.0 ug/ml) intermediate standards in Table 3.1 are also used as spiking standards. For waters, 0.200 ml high level standard is added to the quality control and matrix samples. For soils, 0.800 ml high level standard is added to the quality control and matrix samples.

7.8 Surrogate spiking standard: The surrogate stock standard is diluted in acetonitrile to produce working surrogate solutions of 10.0 ug/mL and 100.0 ug/ml. In the case of samples being tested for Explosives 8330 mix 1 & 2 compounds, ntigrolycerin, and/or PETN, 0.5mL of surrogate solution at 10.0 ug/ml is added to each water sample and water QC sample and 0.2 mL of surrogate solution at 100.0 ug/ml is added to each soil sample and soil QC sample. In the case of samples being tested for nitroguanidine, 0.500 ml surrogate solution at 10.0 ug/ml is added to each water sample and water QC sample, and 0.500 ml surrogate solution at 100.0 ug/ml is added to each soil sample and soil QC sample.

7.9 Calcium Chloride(CaCl2) solution- Add 5.0 grams of anhydrous calcium chloride to a 1.0 Liter volumetric flask and dissolve in DI water. Bring to volume with DI water and invert the volumetric flask to mix.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. The solid samples must be extracted within 14 days and water samples must be extracted within 7 days of the date of collection.

8.2 Sample extracts are stored under refrigeration in the dark and analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

9.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in µg/L and the relative standard deviation (RSD) of the recoveries for each analyte. In addition to each set of IDCs, a blind laboratory spike will be performed. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000, sec. 9.4).

9.3 Louisville Chemistry Guidelines (LCG), require the analysis of method reporting limit (MRL) standards. Programs such as; LCG, DOD Quality Systems Manual (QSM), and Air Force Center for Engineering and the Environment (AFCEE), requires the analysis of a method detection limit (MDL) check standards as another means of checking method performance. The MRLs are analyzed at the beginning and end of each 12 hour shift and are typically prepared at concentrations equal to the reporting limits of the program. Recovery limits are program specific but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x to 3x the method detection limit. MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). MDL check standards are derived from diluting the stock standard to a known concentration of 2x or 3x the method detection limit (Louisville Chemistry Guidelines, LCG). In most instances, a method detection limit check sample is analyzed at the beginning and end of each sequence requiring MRL standards. If the MRL fails for any compound, an MDL must be analyzed immediately following the failed MRL. The recovery criteria for MDL check samples are the ability to detect all compounds at three times the noise level. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

9.4 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SURR, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Programs, client and/or project specific limits are used frequently in sample analysis. Refer to the QSM, DOD, ACOE, LCG, Client QAPPs for program specified limits. In the case where no specified limits are supplied or referred to, in-house limits will be the default limits. The Quality Control Requirements chart also lists specific criteria to the method/project/program.

10.0 CALIBRATION

10.1 Calibration

10.1.1 The initial calibration for SW-846-8330 chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument across two dissimilar analytical columns. Each column must meet the initial calibration and continuing calibration criteria.

10.1.2 The identification of analytes such as explosives is performed by retention times. Retention time windows are crucial to the identification of target compounds. Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in accordance with Method 8000 Section 11.6. Make three injections of the target analyte standards over a course of 72-hour period. Each retention time must be to three decimal places. Calculate the mean and standard deviation of the target analytes. The width is 3x the standard deviation of the mean absolute retention time during the 72 hour period or 0.03 minutes, which ever is higher. If the standard deviation is 0.000, the laboratory will include more data points or use the default value of 0.01 minutes. Each analyte in each standard must fall within its respective retention time window. The retention time shall be set using the midpoint standard of the initial calibration curve or the value in the continuing calibration verification standard at the beginning of the analytical shift. If the analytes fall outside the established retention time window, the HPLC system must either be adjusted so that a second analysis of the standard target analytes fall within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.1.3 For each compound of interest, prepare calibration standards at a minimum of five different concentrations as described above in section 7.4. Note: QSM requires that the LOQ or the lowest point in the curve, which is greater, be used for the reporting limit.

10.1.4 The lowest concentration calibration standard that is analyzed during an initial calibration curve, establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

10.1.5 External standard calibration involves comparison of instrument response from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount of analyte in the calibration standard is defined as the calibration factor (CF).

10.1.6 Linear calibration using the average calibration or response factor: When calculating, both calibration factors and response factors are a measure of the calibration relationship and assume that the curve passed through the origin. If the relative standard deviation (RSD), is less than or equal to 20%, the use of the linear model is generally appropriate, and the calibration curve can be assumed to be linear and to pass through the origin.

10.1.7 Linear calibration using a least squared regression: When the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The approach is to employ a regression equation that does not pass through the origin. This approach can also be employed based on past experience of the instrument response.

10.1.8 Non-linear calibration: When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The statistical considerations in using a non-linear calibration model require more data than the traditional linear approaches. A quadratic (second order) model requires the use of six initial calibration points.

10.1.9 Directly inject the prepared calibration standards into the high pressure liquid chromatograph. An external calibration technique is employed. One of the external standards will be at a concentration near, but above the method detection limit. If the RSD $\leq$ 20%, the correlation of $r \geq 0.995$, or $r^2 \geq 0.990$ is obtained then the calibration is deemed acceptable.

10.1.10 Check the validity of the calibration by analyzing an ICV. The variance of any given compound shall not be more than +/- 15% difference. This standard must be prior to sample analysis. If the percent drift or the percent difference criteria are not met then the subsequent sample analysis for that analyte is not acceptable.

10.1.11 Continuous checking of the validity of the calibration by analyzing CCVs. The variance of any given compound shall not be more than +/- 15% difference. This standard must be ran at the beginning, every 20

samples or every 12-hour interval (which ever comes first), and at the end of the analysis run. Note: QSM samples must have CCVs injected every ten samples for explosives.

10.1.12 It is highly recommended to employ two standards (CCVs) at different concentrations to verify the calibration curve using non-linear calibration. One standard shall be near the quantitation limit or action limit. The choice of specific standards and concentrations is generally a method or project specific consideration.

10.2 Calibration Calculations

10.2.1 When determining Explosives by the external standard technique, calculate the calibration factor (CF) for each peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in Nanograms)}}$$

10.2.2 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation, and the relative standard deviation (RSD) for each multi-component peak.

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (CF_I - \overline{CF})^2}{n-1}\right)}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

10.2.3 Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

10.2.4 The analyst will not force the line through the origin, but have the intercept calculated from the five data points. The use of origin (0,0) as a calibration point: the use of linear regression will not be used as a rationale for reporting results below the calibration range.

10.2.5 A linear least squares regression attempts to construct a linear equation of the form: y= mx + b, by minimizing the differences between the observed results(y_i, the response calculated from the constructed equation). The regression equation is:

$$y_i' = ax_i + b$$

where:

a = Regression coefficient or the slope of the line
b = The y-intercept
y_i' = Predicted (or calculated) response for the ith calibration standard.
x_i = Mass of analyte in the ith calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n} (y_i - y_i')^2$$

where n is the total number of calibration points. The regression calculations attempt to minimize the sum of squares, hence the name "least squares regression."

Weighing the sum of the squares of the differences can significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i (y_i - y_i')^2$$

where:

w_i = Weighting factor for the calibration standard (w=1 for

unweighted least squares regression.

y_i = Observed instrument response (area or height) for the ith calibration standard.

y_i' = Predicted (or calculated) response for the ith calibration standard.

n = Total number of calibration standards.

10.2.6 Least Squares Equation (LSQ) weighting method to be used for calculation of least squares regression fits, either 1/x or $1/x^2$, gives increased importance to smaller concentrations and areas. LSQ Weight can be applied to linear, quadratic, and cubic fits only.

10.2.7 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable, and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it will be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.2.8 The statistical considerations in developing a non-linear calibration model require more data that the more traditional linear approaches described above. Linear regression employ five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards.

10.2.9 The "goodness of fit" of the polynomial equation is evaluated by calculating the weighed coefficient of the determination (COD);

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

.

y_{obs} = Observed response (area) for each concentration of the calibration curve.

$\bar{y}$ = Mean observed response from the initial calibration

Y_I = Calculated response at each concentration from the initial calibrations.
n = Total number of calibration points (6 points for quadratic equation)
p = Number of adjustable parameters in the polynomial.

10.2.10 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0 In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99.

11.0 PROCEDURE

11.1 Water Extraction (Method SW-846, 3535)

11.1.1 Pre-rinse all glassware to be used in the extraction with Acetonitrile (HPLC Grade).

11.1.2 Water samples may be prepared using either the high-level or low-level method. Low-level sample preparation is described below, starting with 11.1.3. To analyze water samples by the high level method, combine 5.0 mL of sample with appropriate volumes of surrogate and/or spiking solutions in a 10 mL volumetric flask. Add acetonitrile to reach a final volume of 10 mL. Mix well and filter the sample through a 0.45-micron filter. Discard the first three mL of filtrate and transfer a portion to a 2.0 mL amber vial for analysis.

11.1.3 From the amber glass sample collection jar, quantitatively measure 500 mL of sample to be extracted (alternatively if sedimentation is not a problem, mark the meniscus on the 500 mL sample jar {if used} so that the volume extracted can be measured after sample extraction). All initial sample volumes need to be noted on a bench sheet in the extraction book.

11.1.4 Clean and rinse (with purified water and acetonitrile) the extraction manifold, its valves, and Teflon samplers, and any other equipment used in the extraction process that can come into contact with the sample.

11.1.5 One method blank (MB) and laboratory control spike (LCS) must be prepared with each batch of 20 samples or less. Prepare by measuring 500 mL of Milli-Q water into a 1 L amber jar.

11.1.6 One sample from each batch of 20 samples or less will be selected for use in the preparation of a matrix spike (MS) and a matrix spike duplicate (MSD). Select the sample and from the glass sample collection jar, quantitatively measure 500 mL of sample to a 1 L amber jar to be spiked and extracted. If insufficient sample was collected to extract an MS/MSD, prepare a duplicate laboratory control spike (LCSD) in order to monitor precision within the extraction procedure.

11.1.7 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike(s), and all samples being tested for explosives 8330 mix 1 & 2 compounds, nitrolycerin and PETN, add 0.5ml of the surrogate standard at 10.0 ug/mL. To the control spike (and possibly the control spike duplicate) and the matrix spike/matrix spike duplicate for explosive 8330 mix 1 & 2 samples add 0.5ml of appropriate spiking standard at 10.0 ug/mL, for NG and PETN samples add 0.2 ml of the appropriate spiking standard at 100 ug/ml.

11.1.8 Assemble the SPE extraction manifold by inserting the disposable Teflon liners into the flow control valves and then close valves. Connect the manifold to a 2 L side arm flask, which is then connected to the in-house vacuum system.

11.1.9 Mount the SPE extraction cartridges onto the flow control valves and condition them using 20-30 mLs of acetonitrile by opening the valves (4 full turns) and letting the acetonitrile pass through the cartridges using gravitational flow into the manifold collection basin (after conditioning transfer the waste acetonitrile into a solvent waste beaker). Be careful not to let the cartridge go dry.

11.1.10 Follow up the acetonitrile conditioning by rinsing the cartridges with 50 mL DI H2O at a flow rate of approximately 10 mL/minute (To obtain the 10 mL /minute flow turn on the house vacuum and set the regulator on the manifold to around 4-5 mm/Hg. Fine adjustments will be necessary). Label each cartridge with the ID of the sample, blank, or spike that is to be extracted. Be careful not to let the cartridge go dry.

11.1.11 Attach the appropriate end of the clean Teflon samplers to the SPE cartridges while placing the weighted end into the sample. When all needed lines are attached, pull the sample, spike, and/or blank through the cartridges by applying the vacuum at a rate of approximately 10 mL/minute. Waste will be collected into the manifold's basin and into the sidearm flask this must be monitored and emptied as needed so the liquid does not get into the vacuum line. If all the positions on the manifold are not being used be sure to close the valves at those positions.

11.1.12 When a sample is completely drawn through a particular cartridge close the valve to that cartridge. When all samples are drawn through their cartridges close all valves and shut off the vacuum. Remove the Teflon samplers from the cartridges and the samples. Empty the manifold basin and the sidearm flask.

11.1.13 Remove the lid to the manifold and place inside the basin a sample tray containing the, appropriately labeled, 15 mL graduated centrifuge (or equivalent) tubes or 10 mL graduated cylinders for collection of the eluted extract.

11.1.14 Elute the samples and their associated QC samples by passing a 5.0 mL aliquot of acetonitrile through the extraction cartridges at about 2 mL/minute (1-2 Hz). Collect the eluate in the centrifuge tubes or graduated cylinders. Record the final volume of eluate collected on the bench sheet.

11.1.15 Prior to analysis, dilute the final extract 50%(v/v) with DI water. Store the final extract in an amber vial, in the dark, and refrigerated until analysis. Mix well and filter the sample through a 0.45-micron filter. Discard the first three mL of filtrate and transfer a portion to a 2.0 mL amber vial for analysis.

11.2 **Solid Extracion (Method SW-846-8330B)**

11.2.1 Prepare solid samples for extraction by first examining the sample matrix for a physical presence of contamination (see Safety, 4.3). Samples will be allowed to air dry at room temperature and in the dark. If determined safe to do so, use a mortar and pestle to homogenize the sample when necessary (To be considered safe, screening by method 8515 can be performed prior to grinding up the sample).

11.2.2 If subsampling is required by the client or project, the entire dried sample must be spread out on a clean surface to a thickness of only 1 to 2 cm and preferably in a fume hood. At least 30 different portions of approximately 0.3 grams each should be obtained from randomly chosen locations by sampling the whole profile.

11.2.3 Weigh 10 grams of sample to the nearest 0.01 g into a 60-mL amber vial and add 20 mL total volume of acetonitrile (included in the volume is that contributed from any surrogate/spiking solution). Note: Wipe samples are directly added to the 60 ml amber sample vial (no sample weight) and brought to volume with 20 mL of acetonitrile. Mix well with vortex mixer. Place the sample(s) into a temperature controlled ultrasonic bath (<30 C) and sonicate for 18 hours.

11.2.4 Prior to extraction add to the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples for explosives 8330 mix 1 & 2, NG, and PETN, 0.2 mL of surrogate solution at 100.0 ug/mL. To the matrix spike/matrix spike duplicate and control spike for explosives 8330 mix 1 & 2 samples, add 1.0 mL of standard spiking solution at 10.0 ug/mL, for NG and PETN, add 0.8 ml of each at 100.0 ug/ml.

11.2.5 One method blank and control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10 grams of organic free silica sand and 20 mL total volume of acetonitrile (surrogate and other spiking solutions volumes included) to a 60-mL amber vial. Mix well and sonicate

for 18 hours in the temperature controlled ultrasonic bath.

11.2.6 One sample from each batch of 20 samples or less must be used in the preparation of a matrix spike (MS) and a matrix spike duplicate (MSD). Weigh 10 grams of sample into a 60-mL amber vial and add 20 mL total volume of acetonitrile (surrogate and other spiking solutions volumes included). Mix well and sonicate for 18 hours in the temperature controlled ultrasonic bath.

11.2.7 After sonication, allow the sample to settle for at least 30 minutes. Remove 5.0 mL of the supernatant and combine it with 5.0 mL of CaCl2 solution in a 20 mL vial, mix and let stand for at least 15 minutes.

11.2.8 Finally, place the supernatant in a disposable syringe, attach a 0.45-micron filter disc, and filter it into the 15 mL amber vials (or directly into 2 mL amber vials used in analysis). Discard the first 2-3 mL of supernatant before adding to the vials. Store the final extract in the dark and keep refrigerated until analysis.

11.3 Water Sample Preparation of Nitroguanidine (NQ) (Reference USACRREL method)

11.3.1 Pre-rinse all glassware to be used in the preparation with acetonitrile (HPLC Grade).

11.3.2 Nitroguanidine water samples are prepared using the high-level method. Combine 5.0 mL of sample with 0.5 mL surrogate at 10.0 ug/mL and in a 10 mL volumetric flask. For the laboratory control (LCS), duplicate laboratory control (LCSD), matrix spike (MS), and duplicate matrix spike (MSD) samples, also add 0.5 mL NQ spike at 10.0 ug/. Add acetonitrile to reach a final volume of 10 mL. Mix well and filter the sample through a 0.45-micron filter. Discard the first three mL of filtrate and transfer a portion to a 2.0 mL amber vial for analysis.

11.4 Soil Extraction of Nitroguanidine (NQ) (Reference USACRREL method)

11.4.1 Pre-rinse all glassware to be used in the extraction with acetonitrile (HPLC Grade).

11.4.2 Prepare solid samples for extraction by first examining the sample matrix for a physical presence of contamination (see Safety, 4.3). Samples will be allowed to air dry at room temperature and in the dark. If determined safe to do so, use a mortar and pestle to homogenize the sample when necessary (To be considered safe, screening by method 8515 can be performed prior to grinding up the sample).

11.4.3 Weigh 2 grams of sample to the nearest 0.1 g into a 60-mL amber vial and

add 50 mL total volume of Millipore water (included in the volume is that contributed from the surrogate; use 0.500 mL high-level surrogate solution at 100.0 ug/mL). Mix well with vortex mixer or shake well for 30 seconds. Place the sample(s) into a temperature controlled ultrasonic bath (<30 C) and sonicate for 2 hours.

11.4.4 One method blank and control spike must be prepared with each batch of 20 samples or less. Prepare by adding 2 grams of organic free silica sand and 50 mL total volume of Millipore water (included in the volume is that contributed from the surrogate; use 0.500 mL high-level surrogate solution at 100.0 ug/mL and 0.500 mL NQ high-level intermediate at 100.0 ug/mL) to a 60-mL amber vial. Mix well for 30 seconds and sonicate for 2 hours in the temperature controlled ultrasonic bath.

11.4.5 One sample from each batch of 20 samples or less must be used in the preparation of a matrix spike (MS) and a matrix spike duplicate (MSD). Weigh 2 grams of sample into a 60-mL amber vial and add 50 mL total volume of Millipore water (included in the volume is that contributed from the surrogate; use 0.500 mL high-level surrogate solution at 100.0 ug/mL and 0.500 mL NQ high-level intermediate at 100.0 ug/mL). Mix well for 30 seconds and sonicate for 2 hours in the temperature controlled ultrasonic bath.

11.4.6 After sonication, allow the sample to settle for at least 30 minutes. Remove 5.0 mL of the supernatant and combine it with 5.0 mL of CaCl2 solution in a 20 mL vial, mix and let stand for at least 15 minutes.

11.4.7 Finally, place the supernatant in a disposable syringe, attach a 0.45-micron filter disc, and filter it into the 15 mL amber vials (or directly into 2 mL amber vials used in analysis). Discard the first 2-3 mL of supernatant before adding to the vials. Store the final extract in the dark and keep refrigerated until analysis.

12.0 SAMPLE ANALYSIS

12.1 Sample Sequence

12.1.1 Before initial calibration or sample analysis, an instrument blank must be analyzed to determine baseline characteristics.

12.1.2 Verify calibration each twelve-hour shift by injecting a Continuing Calibration Verification standard (CCV), prior to conducting any sample analysis. A CCV must be injected at the being of the sequence and at intervals of no less than one per 20 sample injections or twelve hours (whichever comes first) and at the end of each sequence. The variance of any given compound shall not be more than +/- 15% difference. Note:

QSM samples must have CCVs injected every ten samples for explosives.

12.1.3 Before sample analysis, inject a method blank to demonstrate that the instrument, as well as the extraction procedure is free from contamination. All compounds must be below the method detection limit.

12.1.4 Samples can be directly injected after the successful analyses of the calibration curve, CCV, and method blank. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries must be within laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections.

12.1.5 All detects in the associated sample must be confirmed with a second column. Detects are identified and quantitated on the primary column. Results must be confirmed or reported at +/- 40 % on the secondary column. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, then it is be appropriate to report the lower result (method 8000C). Specific projects will require that the higher of the two results be reported. QSM: Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result. Qualify the data with a "Q" flag if target analyte is not confirmed by second column confirmation.

12.1.6 Each sample analysis must be bracketed with passing continuing calibration verification standards. If the continuing calibration verification standard fails to meet QC criteria, all samples that were injected after the last standard that met QC criteria must be re-analyzed.

12.2 Sample Calculations

12.2.1 The computer software determines the concentration of each analyte in the sample. It calculates the amount of compound from the peak area, using the calibration curve. Linear regression of standard responses (R) against their known concentration (C in ug/ml), derives the following equation:

$$C = mR + b$$

12.2.2 Using the slope (m) and the intercept (b) from this equation, the concentration of the sample (ug/ml) is calculated and appears on the report page following the chromatogram.

Water samples:

$$C_S = [(C_C)(V_E)(D)]/V_S$$

Soil samples:

$C_S=[(C_c)(V_E)(D)]/(W)$

Wipe samples:

$C=[(C_c)(V_E)(D)]$

Where: C_S = Concentration of sample in ug/L for waters and mg/kg on a dry weight basis for soils

C_c = Concentration from the curve (ug/ml)

C = Concentration in total ug

V_E = Total volume of sample extract (after concentration) in ml

V_S= Volume of water sample in liters

D = Dilution factor if extract was diluted

W = Weight of dried soil sample in grams

13.0 DATA ASSESSEMENT & ACCEPTANCE CRITERIA FOR QC MEASURES

13.1 Data Assessment & Acceptance Criteria for QC Measures

13.1.1 If the initial analysis of a sample or a dilution of the sample has a concentration of a particular explosive compound that exceeds the calibration range, the sample must be re-analyzed at a dilution.

13.1.2 The qualitative identification of each explosive determined by this method is based on retention time. This method employs a dual column comparison on dissimilar phases. The retention time is updated with the midpoint of each new calibration and is continually updated with the continuing calibration verification standards (CCVs). A new method ID is provided for each new sequence with updated retention times or alteration to the operating initial calibration method. If an alteration occurs (other than daily retention time updates), project management and the QA officer will be notified. A description of the alteration will be addressed in the client notes or case narrative.

13.2 Reporting Quantitative Analysis

13.2.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times are compared and the sample retention times have been made, the sample data can be reported. Assessments of all spiked, calibration control samples, and standards will also be finalized before reporting the data.

13.2.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors, and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 4.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point, the data is turned over to another qualified analyst for final validation. The second analyst confirms the results, electronically marks them validated, and signs his or her portion of the checklist (the checklist is included in this SOP). Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

13.2.3 An electronic copy of the data is then filed and archived. The package includes; the sequence run log, a copy of the bench sheet, the LIMS run log, LIMS prep sheet, verification of calibration data, and each sample, the chromatogram. All the data is to be initialed and dated by the analyst. Each sequence file header will be labeled by the date of sequence.

13.3 Corrective Measures for Out-of-Control Data

13.3.1 When data is out of control, corrective actions are required. Certain situations will cause data deficiencies. If the non-conformities involve failing QC within the analytical sequence batch, the reanalysis of samples will eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument, followed by reanalysis of affected data shall correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument will need cleaning and decontamination, and the sample will need diluting. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions shall be taken for different QC-type failures or non-conformities (Table 3).

13.3.2 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data can be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.

13.3.3 To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises re-analysis or re-extraction is an option.

13.3.4 If a sample is extracted past holding times, the analyst must notify client services immediately. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur. If the client still requests sample analysis, the sample data is qualified for holding time violations.

13.3.5 If the non-conformities involve failing QC within the analytical sequence batch, the analyst must use professional judgment for the reanalysis of the extracts. The reanalysis will ensure that the instrument was functioning properly. If the results are passing, the non-conformities were associated with the instrument. If data is still outside QC criteria, re-extraction of all samples associated with the QC samples must be performed (provide that there is sample available). If no sample is available for re-extraction, the samples are qualified accordingly.

13.3.6 If the out of control data is the result of instrument malfunctions, the analyst must evaluate and repair the instrument prior to reanalysis of the affected data. After the instrument maintenance and repair, the analyst must demonstrate corrective measures by analyzing a Continuing Calibration Verification (CCV) before continuing sample analysis. Any maintenance and repair must be noted in the instrument logbook.

13.3.7 If sample matrix affect or contamination is the reason for poor data. The analyst will use professional judgment for the reanalysis of the extract. The extract can be diluted and reanalyzed. The sample can be re-extracted confirming matrix interference. The analyst can report and qualify the sample data. The analyst must supply explanation for the qualified data in the narrative or client notes.

13.3.8 If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors. Senior analysts have the authority to validate their own data.

13.3.9 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

13.3.9.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

13.3.9.2 In all instances, if results are reported from data that is out of control or unacceptable, that data is qualified accordingly. Once the client has been notified and he or she instructs us to report the data,

the data is qualified to indicating what type of nonconformity has occurred.

13.3.9.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data.

13.3.9.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating the nonconformance and to ensure that the proper corrective actions are employed.

14.0 WASTE MANAGEMENT

14.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).20.1.

15.0 REFERENCES

15.1 USEPA, SW-846, 3rd Ed. Chapters 2&4, Method 8000C. March 2003.

15.2 USEPA, SW-846 Method 8330B. October, 2006.

15.3 USAEC, Methods Manual for Detection of PEP Compounds. EPA Method 8330. May 19, 1999.

15.4 USAEC, Methods for Determining Nitroguanidine in Soil and Water. Special Report 89-35. November 1989.

15.5 Department of Defense, Quality Systems Manual, Rev. 3, January 2006.

15.6 USAEC, Louisville Chemistry Guidelines, June 2002.

Table 3
Summary of Method Quality Objectives for Method 8330- Explosives

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Initial Calibration	Initial Calibration prior to sample analysis. 1. RSD for each analyte $\leq$ 20% 2. Linear – least squares regression r $\geq$ 0.995. 3. Non-linear regression $r^2 \geq 0.99$ (6 points shall be used for second order)	r $\geq$ 0.995, RSD $\leq$20%, $r^2 \geq$0.990, mean RSD $\leq$20%	Linearity must fit one of acceptance criteria, or the curve must be reanalyzed.
Initial Calibration Verification (ICV)	After initial calibration	Difference $\leq$15% QSM: $\leq$20%	If ICV falls outside QC criteria, reanalysis must take place. If ICV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". QSM: No samples will be analyzed until the problem has been corrected.
Continuing Calibration Verification (CCV)	Every twelve hours or every twenty samples (which one comes first) QSM: Every ten samples	Difference $\leq$15% QSM: $\leq$20%	If CCV falls outside QC criteria, reanalysis must take place. If CCV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". Exception to the above, if the acceptance limits are exceeded high and the analyte is not detected in the sample the verification standard has passed (analyte would have been detected if present). QSM: No samples will be analyzed until the problem has been corrected.
Retention Time Window (RTW)	Retention Times will be set using the midpoint standard in the ICAL or the RT in the CCV run at the beginning of each analytical shift.	Shift less than ± 3 times the absolute Standard Deviation from the 72 hour RTW study, with a minimum width of 0.03 minutes	QSM: No samples shall be run without a verified retention time window at the initial verification. For CCVs, apply a "Q" to all results for analytes outside the retention time window.

Table 3
Summary of Method Quality Objectives for Method 8330- Explosives

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
MDL Study/MDL Verification Check	At initial set-up and annually for MDL Study. MDL Verification Checks shall be performed quarterly.	MDL checks must produce a signal at least 3 times the instrument's noise level.	Samples can not be analyzed without a valid MDL. QSM: Run MDL verification check at higher (2x MDL).
MRL Level Verification Check standard at Reporting Limit. (LGG Requirement only)	Beginning and end of 12 hr. sequence or program specified.	70-130% or project specific/client limits	For results > 130% apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at the end and acceptable do not reject data.
Method Blank (MB)	1 per sample batch ≤ 20 samples of the same matrix	Analytes must not he higher than the highest of the following: 1/2 MRL, or 5% of the regulatory limit, or 5% of the associated sample concentration. QSM = ½ MRL	If sample is available and within holding times, sample associated with method blank needs to be reprepped. If no sample is available, qualify the data with a "B" to all associated positives when less than 5X blank concentration. QSM: Apply "B" to all results for the specific analytes in all samples in the associated preparatory batch.
Laboratory Control Sample (LCS)	1 per sample batch ≤ 20 samples of the same matrix	In-house limits, or program or client specified limits	Correct problem, then re-analyze the LCS. If LCS recoveries are still outside QC control limits, then repep and reanalyze the LCS and all samples in the associated batch for the failed analytes in the samples, if sample is available. Qualify the failing analytes with a "Q" qualifier.
Matrix Spike (MS)	1 per sample batch ≤ 20 samples of the same matrix	In-house Limits, or program or client specified limits	No action is taken based on MS recovery alone, use of professional judgement. For recoveries outside QC criteria, qualify out lying compound with "M". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.

Table 3
Summary of Method Quality Objectives for Method 8330- Explosives

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Matrix Spike Duplicate (MSD)	1 per sample batch ≤ 20 samples of the same matrix	In-house Limits, or program or client specified limits	No action is taken based on MSD results alone, use of professional judgement. If RPD is outside QC criteria, then qualify the out lying compounds with "Y". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.
Surrogates	Every sample and QC	In-house Limits, or program or client specified limits	Rerun sample. If no apparent matrix interference noticed re-extract sample. If no sample is available, qualify the surrogate with "S". QSM: For QC and field samples; correct problem, re-analyze, if no apparent interference, re-prep and re-analyze all failed samples or failed surrogates in the associated batch, if sample material is available. If no sample available, field samples apply "J" to the specific sample, QC samples apply "Q" to all associated samples
Target Analyte Confirmation	Whenever a positive is detected on one column, analyzed on a 2nd column.	If present: RPD ≤ 40%	**If not present**; QSM: Report positive value with a "Q" qualifier. Non-QSM: Do not report value. **If present and RPD ≤40%** QSM: Report higher of the two values. Non-QSM: Reporting the lower of the two values may be appropriate. **If present and RPD >40%** QSM: Report higher value with a "J" qualifier. Non-QSM: Report lower of the two values, may be appropriate, with a "P" qualifier

Table 4
8330 Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.

Requirement:	**Acceptance Criteria**	**Analyst Review**		**Independent Review**		**Comments:** (indicate reference to an attachment if necessary)
		Yes	**No**	**Yes**	**No**	
1. INITIAL CALIBRATION (ICAL)						
a. Was the Explosive initial calibration performed using a minimum of five varying standard concentration levels?	Lowest standard at or near MRL					
b. Is the variation between calibration response factors for all concentration levels <20% RSD, is r^2 >.990, or r > 0.995 for the regression line?	RSD <20%, r^2 >.990, r>0.995					
c. Was each ICAL uniquely identified (i.e. Standard Number)?						
d. Was an initial calibration blank (ICB) analyzed?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Was there a second source ICV for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
c. Were the recoveries for the ICV between 85-115% (80-120% for QSM).	% Recovery					
d. Was the ICV uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run?						
b. Were the recoveries for the CCVs within program limits?	%Recovery					
c. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8330 Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was a method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	Per program					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	Per program					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	Per program					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	Per program QSM: 30%RPD					

Table 4
8330 Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
f. Was there a acetonitrile:H2O injection preformed prior to sample analysis?						
9. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the PDF file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

CT Laboratories
Baraboo Laboratory Division

Title: **Flash-Point by Pensky-Martens Closed Cup Tester**

SOP Number:

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

Method 1010 uses the Pensky-Martens closed-cup tester to determine the flash point of liquids including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids shall also be tested using this method.

2.0 METHOD SUMMARY

The sample is heated at a slow, constant rate. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

3.0 DEFINITIONS

3.1 Flash point- the lowest temperature at which application of an ignition source causes the vapors of a specimen of the sample to ignite.

3.1.1 The test specimen is deemed to have flashed when a flame appears and instantaneously propagates itself over the entire surface of the test specimen.

3.1.2 When the ignition source is a test flame, the application of the test flame may cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

3.2 Dynamic- the condition where the vapor above the test specimen and the test specimen are not in temperature equilibrium at the time that the ignition source is applied.

3.3 Equilibrium- the condition where the vapor above the test specimen and the test specimen are at the same temperature at the time the ignition source is applied.

4.0 HEALTH AND SAFETY

4.1 Gloves, lab coat and safety glasses should be worn when performing this test.

4.2 This test should be conducted in a fume hood with the blower off.

4.3 Solvents used in this test are flammable and a health hazard. Keep solvents under the hood.

5.0 CAUTIONS

5.1 *p*-Xylene is flammable and should be handled with care around the open flame.

5.2 All samples should be treated as flammable and handled with care around the open flame.

5.3 For certain mixtures containing halogenated hydrocarbons, no distinct flash is observed. Instead a significant enlargement of the test flame, not halo effect, and change in color of the test flame from blue to yellowish-
orange occurs. Continued heating and testing of these sample above ambient temperature can result in significant burning of vapors outside of the cup and can be a potential fire hazard.

5.4 All solvents are a potential contamination problem for the VOC lab. Keep all solvent bottles closed and under a hood to minimize contamination problems.

6.0 INTERFERENCES

6.1 If the gas test flame is extinguished it will not ignite the vapors in the cup and the gas for the test flame that then enters the vapor space can influence the test result.

6.2 When the ignition source is a test flame, the application of the test flame may cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash point and shall be ignored.

6.3 Remove all cleaning solvents prior to adding sample to the brass cup. Any remaining cleaning solvent can cause an erroneous reading.

6.4 For certain mixtures containing halogenated hydrocarbons, no distinct flash is observed. Instead a significant enlargement of the test flame, not halo effect, and change in color of the test flame from blue to yellowish-orange occurs.

7.0 PERSONNEL QUALIFICATIONS

7.1 Any analyst performing this test should know basic fire safety.

7.2 Analyst must demonstrate proficiency before performing analysis on samples.

8.0 APPARATUS AND MATERIALS

8.1 Precision Scientific Pensky-Martens Flash Point Tester, Semi-Automatic, Model 32140

8.2 Thermometer- Temperature range from 20-230°F.

8.3 Propane Gas Cylinder

8.4 Matches or lighter

8.5 *p*-Xylene- used as LCS

8.6 Toluene, Acetone and Methylene Chloride for use as cleaning solvents when soap and water do not clean the sample cup entirely.

9.0 INSTRUMENT CALIBRATION

9.1 Thermometer must be calibrated in accordance with SOP CL-9.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 Samples should not be collected or stored in gas-permeable containers.

10.2 Caution should be taken to avoid the loss of volatile material.

10.3 Avoid the introduction of moisture to the sample by keeping the lid closed whenever possible.

10.4 Samples should be stored at 4^{0}C.

10.5 Do not transfer the sample into the brass testing cup until the sample temperature is at least 18^{0}F below the expected flash point.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Place flash point apparatus into hood and turn off exhaust fan of hood.

11.2 Fill the brass test cup to line with sample or LCS, whichever is to be tested.

11.3 Place the brass test cup into the flash point apparatus and secure the stirrer top.

11.4 Turn stirrer on but only connect the stirrer to the cup apparatus if the sample is liquid.

11.5 Light test flame. Adjust flame to 3.2-4.8mm diameter

11.6 Lower test flame into cup to see if sample ignites

11.7 If sample does not ignite, turn heater so that the temperature increases 10^{0}F/minute.

11.8 Apply the test flame when the temperature is 40° F below the expected flash point. If flash point is unknown, start at room temperature (60 ± 10° F).

11.9 Continue applying test flame every 5° F until flash point is seen or you have passed 140° F.

11.10 If a flash is seen, discard the sample and reanalyze a fresh portion of sample. Begin applying flame at 41 $\pm$ 9^0F below temperature of flash point from the initial analysis.

11.11 Report flash point as the temperature where you saw the flash point or as >140° F if no flash point was seen before the temperature exceeded 140° F.

11.12 After sample cup and flash point apparatus have cooled, remove the sample cup and dispose of the sample.

11.13 Clean the cup with soap and water. Use a cleaning solvent to remove any residue not removed by soap and water.

11.14 Dry cup thoroughly.

12.0 TROUBLESHOOTING

Consult the Precision Scientific manual for troubleshooting instructions.

13.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

13.1 Data is manually acquired by recording the temperature of the flash point on a lab bench sheet.

13.2 The data is manually entered into the LIMS system by the analyst.

13.3 There are no special calculations required for flash point determination.

13.4 Data reduction is not required for flash point determination.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 A computer with LIMS software.

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 After the data has been entered into the LIMS system by the analyst, it is reviewed by another qualified analyst or the inorganic group leader

15.2 After reviewing the data in LIMS, if the data is deemed to be valid, then the reviewer changes the status of the batch in LIMS to validated.

15.3 The data is stored in the inorganic data file cabinet and the data in the cabinet is archived in the warehouse as needed.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 An LCS consisting of *p*-Xylene should be run every 20 samples per day. The flash point should be between 77.9-81.9^0F.

16.2 A duplicate sample should be analyzed every 20 samples per matrix per day. The duplicate flash point should be within 5^0F. If the duplicate sample does not fall within the acceptable range, the analysis should be repeated.

17.0 REFERENCES

6.1 *Test Methods for Evaluating Solid Waste*, EPA, 1986, SW-846, 3rd Edition, Rev 0, September 1986, Method 1010.

6.2 *Annual Book of ASTM Standards*, ASTM, 2000, October 2000, Method D 93-00.

6.3 Precision Scientific Pensky-Martens Flash Point Tester Semi-Automatic Manual.

Data Validation Checklist

LIMS #:	Method: Flashpoint EPA Method 1010			
Analysis Date	Analyst / Data Interpretor	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Was LCS within acceptable limits?	77.9-81.9 F					
2. Were duplicates analyzed at the a ppropriate frequency	1 every 20 samples of the same matrix					
3. Were the duplicates within acceptable limits?	Differ by ≤ 5 F					
2. Are all samples on the job lists accounted for?	---					

Hexavalent Chromium, Automated Colorimetric

Approved by: _____
Laboratory Director

Reviewed by: _____
Quality Assurance

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine Cr(VI) in surface waters, drinking waters, domestic wastewaters and industrial wastewaters. The applicable range is 0.02 to 0.40 mg/L. This range may be extended by sample dilution.

2.0 REFERENCES

2.1 *Standard Methods for the Examination of Water and Wastewater*, 18th Edition 1992, APHA-AWWA-WEF, Method 3500-Cr D.

2.2 Lachat QuikChem Methods Manual, 1993. Method Number 10-124-13-1-A.

2.3 Test Methods for Evaluating Solid Wastes, SW 846, Third Edition, July 1992, Method 7196A.

3.0 INTERFERENCES

3.1 Substances which can reduce Cr(VI) upon acidification (e.g. cyanides, thiosulfate, organic matter) will cause negative interferences in the determination of Cr(VI).

3.2 Hexavalent molybdenum and mercury salts will react to form color with the reagent but the intensities are much lower than that for chromium at the specified pH. Concentrations as high as 200 mg Mo or Hg/L can be tolerated.

3.3 Vandadium interferes strongly but concentrations up to 10 times that of chromium will not cause trouble.

3.4 Iron in concentrations greater than 1 mg/L may produce a yellow color but the ferric ion (Fe^{+3}) color in not strong and no difficulty is encountered normally if the absorbance is measured photometrically at the appropriate wavelength.

4.0 DEFINITIONS

4.1 Initial Calibration Verification (ICV): A second source (independently prepared from the calibration standards), mid-level standard analyzed immediately after the calibration, to evaluate instrument performance.

4.2 Initial Calibration Blank (ICB): An aliquot of reagent water analyzed immediately after the ICV. The ICB is used to determine if the method analyte or other interferences are present in the laboratory environment, the reagents or analytical system. Note: Since this method does not involve a preparation step, such as digestion, an ICB takes the place of a method blank (MB).

4.3 Lab Control Sample (LCS): An aliquot of reagent water to which known quantities of method analyte is added. The LCS is analyzed exactly like a sample, and its purpose is to determine whether method performance is within acceptable performance limits.

4.4 Continuing Calibration Verification (CCV): A check standard analyzed after every ten samples and at the end of the analytical run, to evaluate instrument performance.

4.5 Continuing Calibration Blank (CCB): An aliquot of reagent water analyzed immediately after the CCV. The CCB is used to determine if the method analyte or other interferences are present in the laboratory environment, the reagents or analytical system.

4.6 Matrix spike-matrix spike duplicate (MS-MSD): To two of three aliquots of a given sample a known amount of spike solution is added. Matrix spikes and matrix spike duplicates are taken through all steps of preparation and analysis, exactly like a sample. The purpose of the MS-MSD is to determine whether the sample matrix contributes precision or bias to the analytical result. An MS-MSD should be prepared for every 20 samples in each matrix. Note: In situations where the concentration of the sample is greater than 4 times the spike concentration the MS-MSD may be used as a method duplicate (MD). The precision of the MD is determined by calculation the relative percent difference (RPD) (10.3) of the two samples.

5.0 APPARATUS AND MATERIALS

5.1 Class A volumetric flasks and pipets.

5.2 Drying oven capable of maintaining a temperature of 104 ± 1 º C for 2 hours - Fisher Isotemp or equivalent.

5.3 Analytical balance, capable of measuring to 0.0001g - Fisher Scientific A-200DS or equivalent.

5.4 NIST traceable Class 1 calibration weights - 0.002-100g.

5.5 Lachat QuikChem IV (or equivalent) flow injection ion analyzer with a 540 nm filter.

6.0 **REAGENTS**

6.1 Deionized water (DI). The DI water should be monitored for impurities.

6.2 Methanol, HPLC grade, EM Science part #MX0482-6 or equivalent.

6.3 Color Reagent: in a 1 L volumetric flask, dissolve 0.40 g of s-diphenylcarbazide (1, 5–dephenylcarbohydrazide, 2 2 – dephenycarbonic dihydazide, $C_{13}H_{14}N_4O$), ACS reagent grade (Sigma part #D-7766 or equivalent), in 200 mL methanol. Add 720 mL of deionized water, then 80.0 mL of concentrated sulfuric acid (Fisher part #. Dilute to mark with DI water. Storage: room temperature. Shelf life: one month.

6.4 Calibration Stock Standard, 1000 mg Cr/L as K_2CrO_4: Dissolve 2.829 g of dried (105 º C) $K_2Cr_2O_7$ (Fisher part # P188-500 or equivalent) in reagent water in a 1 L volumetric flask and dilute to the mark. Alternatively, a 1000mg/L Cr(VI) certified primary standard solution may be purchased. Storage: room temperature. Shelf life: one year or manufacturer specified.

6.5 Calibration Intermediate Stock Standard, 10.0 mg/L as K_2CrO_4: using the 1000 mg/L standard above pipet 1.00 mL 1000 mg/L standard into a 100 mL volumetric and bring up to volume with reagent water. Storage: room temperature. Shelf life: one year.

6.6 Calibration Working standards: into 5 – 250 mL volumetric flasks pipet, 10.0, 5.0, 2.5, 1.25, & 0.5 mL of the intermediate stock standard and dilute to volume with DI water. Invert three times. This yields standards of 400, 200, 100, 50, and 20 ug Cr/L respectively. Storage: room temperature. Shelf life: one year.

6.7 Calibration Verification Stock Standard, 1000 mg Cr/L as K_2CrO_4: Dissolve 2.829 g of dried (105 º C) $K_2Cr_2O_7$ (Fisher part # P188-500 or equivalent) in reagent water in a 1 L volumetric flask and dilute to the mark. Alternatively, a 1000mg/L Cr(VI) certified primary standard solution may be purchased. **Note:** This must be prepared separately from the calibration stock standard, in order to serve as a second source. Storage: room temperature. Shelf life: one year.

6.8 Calibration Verification Intermediate Stock Standard, 10.0 mg/L as K_2CrO_4: using the 1000 mg/L calibration verification standard prepared above, pipet 1.00 mL 1000 mg/L standard into a 100 mL volumetric and bring up to volume with reagent water. Storage: room temperature. Shelf life: one year.

6.9 Initial Calibration Verification Standard, (ICV) 300 ug/L: into a 250 mL volumetric flask pipet 7.5 mL of calibration verification intermediate stock standard and dilute to volume with DI water. Storage: room temperature. Shelf life: one year.

6.10 Laboratory Control Sample: 0.1 – 1.0 mg/L. NIST Traceable. Analytical Products Group, Catalog # 7745 or equivalent. Storage: room temperature. Shelf life: manufacturer specified.

7.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Collect a minimum of 125 mL of sample in plastic or glass containers.

7.2 Samples should be kept at 4 ± 2 ºC without chemical preservative until analysis. Hold time is 24 hours.

8.0 PROCEDURE

8.1 Refer to Lachat manual for a diagram of the manifold. Set up program to the following settings:

Injection timing:

Pump speed:	35
Cycle speed:	50s
Inject to peak start period:	14s
Sample loop length:	120 cm
Filter:	540nm

8.2 Inspect modules for proper connections.

8.3 Turn on power to all modules.

8.4 Place reagent lines into proper containers. Raise tension on pump tube cassettes.

8.5 Initiate and pump system until stable, as indicated by monitoring the baseline. A stable baseline is defined as no visible change (without fluctuation) - in other words a visually straight line indicates the system is stable.

8.6 Set zero on colorimeter.

8.7 Place calibration standards in sample tray in descending order of concentration. Analyze and develop a standard curve based on plotting absorbance against the known concentrations. The concentrations of the unknowns (samples), and QC, are evaluated in relation to the absorbencies of the calibration standards. The Lachat software program prints out the raw data in ug/L.

8.8 Verify the acceptance of the calibration curve by analyzing the ICV standard.

8.9 Verify the system is free of interferences by analyzing a ICB.

8.10 If items 8.7 through 8.9 meet acceptance criteria (see Section 9) samples, LCS and MS-MSD can be analyzed in groups of 20 or less samples, per similar matrix. The LCS is prepared according to manufacturer instructions and analyzed in the same manner as samples. MS-MSD is prepared by choosing a sample within the group at random, transferring two 3-mL aliquots into autosampler containers and fortifying with 1-mL of the 1000 ug/L calibration intermediate stock standard (6.5).

8.11 The calibration curve and system acceptability is checked after every 10 or less analysis, and at the end of the analytical sequence, by analyzing a CCS and a CCB.

8.12 Immediately at the end of the quantitation run begin a turbidity check on positive samples. Remove the diphenylcarbazide transmission line (from the color reagent) and place it in DI water. Allow the system to pump for one minute. Reanalyze all samples with a concentration > 0.02 mg/L. Correct the sample initial reading by subtracting the sample turbidity reading.

8.13 At the end of the run place all transmission lines in water, and flush system. Pump lines dry.

8.14 Turn off pump, all modules, and release tension levers on pump tube cassettes.

9.0 QUALITY CONTROL

9.1 The analyst must show an initial demonstration of capability (IDC) to generate acceptable data, by successfully analyzing four aliquots of reagent water to which know quantities of analyte is added. Acceptance criteria for accuracy is 80-120% and 20% RSD for precision.

9.2 ICV (initial calibration verification): This mid range second source check standard is analyzed after calibration. Control limits are +/- 10% of the expected concentration. If the recovery is outside of this range, terminate the run and correct the problem before proceeding.

9.3 ICB (initial calibration blank): Analyzed immediately after the ICV. The concentration of the ICB must be below 0.02 mg/L. If not, terminate the analysis and correct the problem before proceeding.

9.4 LCS (lab control sample): Analyze one LCS per 20 samples. Control limits are +/- 10% of the expected concentration. If the control limit is exceeded, terminate the analysis, correct the problem and reanalyze all samples back to the last acceptable LCS.

9.5 CCV (continuing calibration verification): A CCV mid-range second source check standard is analyzed following every ten samples and at the end of the analysis. Control limits are +/- 10% of the expected concentration. If recovery is outside these limits, remake and reanalyze CCV once, if still unacceptable, recalibrate or reanalyze all samples back to the last acceptable CCV or ICV.

9.6 CCB (continuing calibration blank): Analyzed immediately after a CCV. The concentration of the CCB must be below 0.02 mg/L. If the CCB is above this level, reanalyze CCB once. If still unacceptable, terminate the analysis, correct the problem, recalibrate, and reanalyze all samples back to the last acceptable CCB, initiating the reanalysis with a CCV or ICV..

9.7 MS-MSD (matrix spike-matrix spike duplicate): A MS-MSD is required every analytical run at a frequency of 5% (1 set for every 20 samples) per matrix type. Acceptance criteria for both accuracy and precision is 80-120% for accuracy and 20% RPD for precision. The spiking level should be 2 to 5 times the spiked sample concentration or within the upper half of the calibration curve.

9.8 MD (method duplicate): If a MS-MSD is not applicable (native concentration > 4X spike level) the MS-MSD functions as a MD, at the frequency is 1 per 20 samples. The acceptance criteria is a RPD of $\leq$ 20%.

PROPRIETARY

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Five-point initial calibration	Initially and as needed	$r \geq 0.995$ for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R: 90-110%	Remake and reanalyze ICV standard once, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After ICV, prior to sample analysis	< 0.02 mg/L	Remake and reanalyze CB once, if unacceptable investigate and correct problem. Reanalyze along with associated samples.
Continuing calibration verification (CCV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R: 90-110%	Reanalyze CCV once, if unacceptable investigate and correct problem. Reanalyze all samples after last acceptable CCV.
Continuing calibration blank (CCB)	After each CCV	< 0.02 mg/L	Halt operation, fix problem and reanalyze all samples back to the last acceptable CCB
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	%R: 80-120% RSD $\leq$ 20%	Retrain and repeat as needed until acceptable
Laboratory control sample (LCS)	One (1) per of 20 samples of the same matrix	80% $\leq$ %R $\leq$ 120%	Reanalyze once, if unacceptable investigate and correct problem. Reanalyze along with associated samples.
Matrix spike sample (MS)	One (1) per of 20 samples of the same matrix	75% $\leq$ %R $\leq$ 125%	Investigate problem, if LCS in control qualify results as matrix interference.
Matrix spike duplicate (MSD) or Method duplicate (MD)	One (1) per of 20 samples of the same matrix	RPD $\leq$ 20%	Investigate problem, if LCS in control qualify results.

10.0 CALCULATIONS

10.1 Accuracy- MS-MSD

$$\textit{Spike Concentration, ug/L} = \frac{\textit{(mLs of spike used) (conc. of spiking soln, ug/L)}}{\textit{(combined vol. of sample + spike, mL)}}$$

$$\textit{\% Spike Recovery} = \frac{\textit{[spike + sample] - [sample x C.F.*]] x 100}}{\textit{expected concentration}}$$

$$\textit{*C.F (volume correction factor)} = \frac{\textit{Volume of sample, mLs}}{\textit{vol. of sample + spike, mLs}}$$

10.2 Accuracy- ICV and CCV

$$\textit{\% Recovery of Standard} = \frac{\textit{measured concentration x 100}}{\textit{expected concentration}}$$

10.3 Precision- Relative percent difference (RPD*):*

$$\textit{\% RPD} = \frac{\textit{Abs.** [sample conc. - dup conc.] x 100}}{\textit{ave. of sample \& dup conc.}}$$

***Absolute value*

11.0 SAFETY

11.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTI Health and Safety Manual).

Hexavalent Chromium, Alkaline Digestion Followed by Colormetric Analysis

Approved by: _____
Laboratory Director

Reviewed by: _____
Quality Assurance

1.0 SCOPE AND APPLICATION

1.1 This method is for the determination of hexavalent chromium in soil, sludges, sediments, and similar waste materials. The applicable range is 0.52 to 8.0 mg/kg. This range may be extended by sample dilution.

1.2 In order to determine the concentration of hexavalent chromium in a solid sample, samples must be extracted by an alkaline digestion procedure. The extract is then analyzed colorimetrically, at 540nm, on a spectrometer.

2.0 REFERENCES

2.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, July 1992, Method 3060A.

2.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, July 1992, Method 7196A.

3.0 INTERFERENCES

3.1 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

3.2 For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(IV) results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phoshate buffer to the alkaline extraction solution has been shown to suppress this oxidation.

3.3 Iron in concentrations greater than 1 mg/L may produce a yellow color but the ferric ion (Fe^{+3}) color in not strong and no difficulty is encountered normally if the absorbance is measured photometrically at the appropriate wavelength.

4.0 DEFINITIONS

4.1 Initial Calibration Verification (ICV): A second source, midrange standard, taken through the same digestion as the samples and analyzed at the beginning of a run, to evaluate calibration curve and or instrument performance.

4.2 Method Blank (MB): Analyte free Ottawa sand taken through the same digestion and analytical process as the samples. The MB is used to document contamination in the analytical process.

4.3 Lab Control Sample (LCS): Analyte free Ottawa sand spiked with the target analyte. A LCS is used to document laboratory performance.

4.4 Continuing Calibration Verification (CCV): A second source, midrange check standard analyzed after every ten samples and at the end of the analytical run, to evaluate instrument performance. The CCV standard is taken through the digestion and analytical process as the samples.

4.5 Matrix Duplicate (MD): A split sample, which is used to document the precision in a given matrix.

4.6 Soluble Matrix Spike: An aliquot of sample spiked with a known concentration of analyte. The spiking of the soluble matrix spike occurs prior to sample digestion. A soluble matrix spike is used to document the bias of the method in a given matrix.

4.7 Insoluble Matrix Spike: An aliquot of sample spiked with a known concentration of the solid matrix spiking agent $PbCrO_4$ (6.7). The spiking of the insoluble matrix spike occurs prior to sample digestion. An insoluble matrix spike is used to document the dissolution during the digestion process in a given matrix.

4.8 Post-digestion Matrix Spike (PDS): An aliquot of sample digestate spiked with a known concentration of analyte. The PDS occurs after sample digestion. A PDS is used to document the bias arising from the digestion process on a given matrix.

5.0 APPARATUS AND MATERIALS

5.1 Analytical balance, Fisher Model A-200DS or equivalent. Calibrated with NIST traceable Class1 weights ranging from 0.002-100g.

5.2 Boiling chips Chemware or other Teflon brand

5.3 Digestion cups with snap caps, Environmental Express, Part # SC500

5.4 Block digestion system, Environmental Express, Part #SC100

5.5 Traceable brand, NIST traceable thermometer or equivalent

5.6 pH meter, Orion Model 920A or equivalent

5.7 Volumetric pipets, Class A

5.8 VWR brand stir plate and stir bars, Model #205, or equivalent

5.9 Volumetric flasks, Class A

5.10 Spectrophotometer, Milton Roy Model Spectronic 401 or equivalent

5.11 Sterile, disposable polystyrene vacuum filtration apparatus with 0.45um cellulose membrane filters, Corning Brand, part #430947

5.12 Graduated cylinders, 50 mL TD

6.0 REAGENTS

6.1 Prepare all standards and solutions using deionized water (DI).

6.2 Concentrated o-Phosphoric Acid (85%), ACS reagent grade. Fisher part #A242^{SK}-212 or equivalent.

6.3 Sodium Carbonate: Na_2CO_3 anhydrous, ACS reagent grade. Fisher part #S263-500 or equivalent.

6.4 Sodium Hydroxide: NaOH, ACS reagent grade. VWR part #VW6720-5 or equivalent

6.5 Magnesium Chloride: $MgCl_2$, ACS analytical grade. (A mass of 400 mg $MgCl_2$ is approximately equivalent to 100 mg Mg^{2+}). Sigma part #M-8266 or equivalent.

6.6 Phosphate Buffer:

6.6.1 K_2HPO_4: ACS reagent grade. EM Science #PX1570-1

6.6.2 KH_2PO_4: ACS reagent grade. Fisher part #P285-500

6.6.3 **0.5M K_2HPO_4 / KH_2PO_4 buffer at pH 7**: Into a 1 L volumetric flask, dissolve 87.09g K_2HPO_4 and 68.04 g KH_2PO_4 and bring up to volume with reagent water. Storage: room temperature. Shelf life: one year.

6.7 Lead Chromate: $PbCrO_4$, analytical reagent grade. Store under dry conditions at 20-25 º C in a tightly sealed container. JT Baker Part #2314-4

6.8 Digestion Solution: Dissolve 20.0 $\pm$ 0.05 g NaOH and 30.0 $\pm$ 0.05 $NaCO_3$ in reagent water in a 1 L volumetric flask and dilute to the mark. The pH must be checked daily before using, pH $\geq$11.5, if not, discard. Storage: room temperature. Shelf life: one year.

6.9 Potassium dichromate Stock Solution, $K_2Cr_2O_7$, stock standard (1000 mg/L Cr(VI) *for calibration use only*: Dissolve 2.829 g of dried (105 ° C) $K_2Cr_2O_7$ (Fisher part # P188-500 or equivalent) in reagent water in a 1 L volumetric flask and dilute to the mark. Alternatively, a 1000mg/L Cr(VI) certified primary standard solution may be purchased. Storage: room temperature. Shelf life: one year or manufacturer specified.

6.10 Intermediate Stock Standard, 10 mg/L Cr(VI) *for calibrtion use only*: using the 1000 mg/L standard above (6.9) pipet 1.00 mL 1000 mg/L standard into a 100 mL volumetric and bring up to volume with reagent water. Storage: room temperature. Shelf life: one year.

6.11 Calibration Standards: Into 2-50 mL digestion vessels, pipet 0.02 mL and 0.01 mL of 1000 mg/L Cr(VI) (6.9) and follow steps below (8.1-8.16), yielding a 200 ug/L and 100 ug/L standard respectively. Then into 3-50 mL digestion vessels, pipet 0.5, 0.25, and 0.1 mL respectively of the 10 mg/L Cr(VI) (6.10) and carry through steps 8.1-8.16, yielding a 50, 25 and 10 ug/L standard respectively. Prepare fresh as needed.

6.12 Potassium dichromate Calibration Verification and Soluble Spiking Solution, $K_2Cr_2O_7$, stock standard (1000 mg/L Cr(VI) *prepare independent from (6.9)*: Dissolve 2.829 g of dried (105 ° C) $K_2Cr_2O_7$ (Fisher part # P188-500 or equivalent) in reagent water in a 1 L volumetric flask and dilute to the mark. Alternatively, a 1000mg/L Cr(VI) certified primary standard solution may be purchased. Storage: room temperature. Shelf life: one year or manufacturer specified.

6.13 Intermediate Calibration Verification and Soluble Spiking Solution, 10 mg/L Cr(VI) *prepare independent from (6.9)*: using the 1000 mg/L standard above (6.12) pipet 1.00 mL 1000 mg/L standard into a 100 mL volumetric and bring up to volume with reagent water. Storage: room temperature. Shelf life: one year.

6.14 Calibration Verification Standard, 50 ug/L: Into a 50 mL digestion vessel, pipet 0.5 mL of the 10mg/L independently prepared Cr(VI) standard (6.13) and take through steps 8.3-8.16, as outlined below.

6.15 Diphenylcarbazide solution: Dissolve 0.25 g of 1,5-diphenylcarbazide (Sigma part #D-7766) in 50 mL of HPLC grade methanol (EM Science part #MX0482-6). Storage: room temperature in an amber bottle. Prepare fresh daily.

7.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Samples should be collected using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass).

7.2 Samples should be stored field-moist at 4 ± 2º C until analysis.

7.3 Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection. In addition, Cr(VI) has also been shown to be stable in the alkaline digestate for up to 168 hours (7 days) after extraction from the soil matrix

8.0 PROCEDURE

8.1 Adjust the temperature setting on the Hot Block digestion system to 90-95 º C and monitor the temperature using a NIST-traceable thermometer.

8.2 Place 2.5 ± 0.1 g of well mixed, field-moist sample into a 50 mL digestion cup. For the specific sample(s) that is/are being spiked, the sample material should be added at this time (8.2.1 ; 8.2.2)

8.2.1 Soluble matrix spike: Add 1.0 mL of the 10 mg/L spike solution (6.10) should be added directly to the sample aliquot. (Note: Spike concentrations can be adjusted as needed due to background).

8.2.2 Insoluble matrix spike: To a separate sample aliquot, all 0.01-0.02 g of $PbCrO_4$ (6.7).

8.3 Add 50 mL ± 1 mL of digestion solution (6.8) to each sample using a graduated cylinder.

8.4 Add approximately 0.4 g of $MgCl_2$ (6.5) to each sample cup.

8.5 Pipet 0.5 mL of 1.0 M phosphate buffer (6.6) to each digestion cup, add a stir bar and cover with snap caps.

8.6 Stir samples continuously for at least five minutes.

8.7 Remove caps from the sample cups, place 4-6 boiling chips in each vessel, and place the cups in a digestion rack. Place the rack on a preheated (to 90-95 º C) digestion block and allow the samples to digest at this temperature for at least 60 minutes.

8.8 Remove the rack from the digestion block and allow samples to cool to room temperature.

8.9 Quantitatively transfer the contents of each vessel to a filter apparatus. Remove all residual, by rinsing the digestion cup with 3 successive aliquots of reagent water and adding these rinsates to the filtration apparatus (5.11).

8.10 Transfer the filtrate and rinses to a clean 100 mL volumetric flask and bring up to volume with reagent water. Once volume is adjusted to 100 mls, pour off 50 mls of this sample into the original digestion vessel and cap

8.11 Place a stir bar into each digestion vessel and place the vessel on a stir plate. (The following color development steps must be performed in a hood.)

8.12 With continual stirring, slowly add concentrate o-Phosphoric acid (6.2) until pH of 2 ± 0.5. The samples will effervesce, so add the acid SLOWLY.

8.13 Once the pH of the sample is at 2 $\pm$ 0.5, add 2.0 mls of the Diphenylcarbazide solution (6.15). Continue to stir until the effervescence becomes minimal.

8.14 Transfer this sample to a 100 mL volumetric flask and bring up to volume with reagent water. Note: If this solution is turbid after dilution to 100 mL prepare a sample with all reagents except the carbazide reagent and take an absorbance reading using this sample. The absorbance reading obtained from this sample will then be used to correct the absorbance reading of the final colored solution by subtracting the absorbance measured previously.

8.15 Allow color to develop for 5-10 minutes.

8.16 Transfer a portion of the color developed sample to a cuvett and measure at 540 nm on a spectrophotometer. Using a curve developed as described below (8.17), the concentrations of the samples will be measured in ug/L of Cr(VI).

8.17 Preparation of the calibration curve:

8.17.1 To compensate for possible slight losses of chromium during the digestion process or other operations of the analysis, treat the chromium standards by the same procedure as the samples, steps 8.1-8.16. Prepare the standards following 6.11.

8.17.2 Once the standards are color developed, measure the absorbencies, at 540 nm, from low standard to high standard, following the calibration instructions as appears in the Milton Roy Operator's Manual pg. 6-26 section 6.10.2. The correlation coefficient must be $\geq$ 0.995

9.0 QUALITY CONTROL

9.1 The analyst must show an initial demonstration of capability (IDC) to generated acceptable data, by successfully analyzing four replicate spike samples at 4.0 mg/kg (1 mL of soluble matrix spiking solution). Acceptance criteria for accuracy is 80-120% and 20% RSD for precision.

9.2 ICV (initial calibration verification): This mid-range second source standard analyzed immediately after calibration. Control limits are +/- 10% of the expected value. If the recovery is outside of this range, terminate the run, correct the problem before proceeding.

9.3 CCV (continuing calibration verification): Analyze a CCV following every ten samples, and at the end of the analysis. Control limits are +/- 10% of the expected concentration. If recovery is outside acceptable limits, recalibrate and reanalyze all samples back to the last acceptable CCV.

9.4 MB (method blank): The concentration of the MB must be below 10 ug/L. If not, terminate the analysis and correct the problem before proceeding. If the associated samples are affected, redigest/reanalyze all associated samples.

9.5 LCS (lab control sample): Analyze one LCS per group of 20 or less samples of similar matrix. The acceptable range for the LCS is 80-120%, spiked at 4.0 mg/kg (1 mL of soluble matrix spiking solution). If the recovery exceeds this, terminate the analysis, correct the problem and redigest/reanalyze all associated samples.

9.6 Soluble matrix spike is required per group of 20 or less samples of a similar matrix. The acceptable range for soluble matrix spike is 75-125%, spiked at 4.0 mg/kg (1 mL of soluble matrix spiking solution).

9.7 Insoluble matrix spike is required per group of 20 or less samples of a similar matrix. The acceptable range for soluble matrix spike is 75-125%, spiked with 10 – 20 mg of $PbCrO_4$.

9.8 MD (Matrix duplicate): A MD is run at the frequency of 1 required per group of 20 or less samples of a similar matrix. The acceptable range for the MD is a RPD of $\leq$ 20% if sample result is >2.08 mg/kg, or ± 0.52 mg/kg if sample result is $\leq$2.08 mg/kg.

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Five-point initial calibration	Annually	r ≥ 0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	At the beginning of each run.	%R: 90-110%	Reanalyze once, if unacceptable repeat color development step or redigest and reanalyze.
Method blank (MB)	After ICV, prior to sample analysis	< 10 ug/L	Reanalyze once, if unacceptable investigate and correct problem. Redigest along with associated samples and reanalyze.
Continuing calibration verification (CCV)	After every 10 samples, and at end of run	%R: 90-110%	Reanalyze once, if unacceptable, investigate and correct problem. Reanalyze all samples after last acceptable CCV.
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	%R: 80-120% RSD ≤ 20%	Retrain and repeat as needed until acceptable
Matrix duplicate (MD)	One (1) per analytical batch per of 20 or less samples per matrix	RPD ≤ 20% if matrix > 2.08 mg/kg, ± 0.52 mg/kg if matrix < 2.08 mg/kg.	Investigate problem; if LCS is in control qualify results.
Laboratory control sample (LCS)	One (1) per analytical batch per of 20 or less samples per matrix	80% ≤ %R ≤ 120%	Reanalyze, if still outside acceptance, redigest and reanalyze along with associated samples.
Soluble matrix spike	One (1) per analytical batch per of 20 or less samples per matrix	75% ≤ %R ≤ 125%	Reanalyze once, if LCS in control quantify results as matrix interference.
Insoluble matrix spike	One (1) per analytical batch per of 20 or less samples per matrix	75% ≤ %R ≤ 125%	Reanalyze once, if LCS in control quantify results as matrix interference.
Post-digestion spike (PDS)	One (1) per analytical batch per of 20 or less samples per matrix	85% ≤ %R ≤ 115%	Reanalyze once, if still outside acceptance, qualify results.

PROPRIETARY

10.0 CALCULATIONS

10.1 Accuracy- MS-MSD

$$\textit{Spike Concentration, ug/L} = \frac{\textit{(mLs of spike used) (conc. of spiking soln, ug/L)}}{\textit{(combined vol. of sample + spike, mL)}}$$

$$\textit{\% Spike Recovery} = \frac{\textit{[spike sample result] - [sample x C.F.*] x 100}}{\textit{spike added}}$$

$$\textit{*C.F (volume correction factor)} = \frac{\textit{Volume of sample, mLs}}{\textit{vol. of sample + spike, mLs}}$$

10.2 Accuracy- ICV and CCV

$$\textit{\% Recovery of Standard} = \frac{\textit{measured concentration}}{\textit{expected concentration}} \quad X \quad 100$$

10.3 Precision- Relative percent difference (RPD*):*

$$\textit{\% RPD} = \frac{\textit{Abs.** [sample conc. - dup conc.] x 100}}{\textit{ave. of sample \& dup conc.}}$$

***Absolute Value*

10.4 Concentration of sample (Dry weight basis):

$$\textit{Conc mg/kg} = \frac{\textit{Sample concentration (ug/L) x D x final volume (mL) x 100}}{\textit{Grams of sample x percent solids x 1000}}$$

D*=Dilution factor*

10.5 Concentration of sample (As is basis):

$$\textit{Conc mg/kg} = \frac{\textit{Sample concentration (ug/L) x D x final volume (mL)}}{\textit{Grams of sample x 1000}}$$

D*=Dilution factor*

11.0 SAFETY

11.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTI Health and Safety Manual).

CT Laboratories
Baraboo Laboratory Divison

Title: **Inductively Coupled Plasma Emission—ICP-OES 6000 Series**

SOP Number:

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 Identification of Test Method

1.1 This procedure is used for the analysis of trace elements (metals) following EPA SW 846 Methods 6010B (Inductively Coupled Plasma –Atomic Emission Spectrometry) and Method 200.7 (SDWA).

2.0 Applicable Matrix or Matrixes

2.1 This method is applicable to the determination of various metals in drinking water, surface water, groundwater, sludge, soils, and industrial wastes.

3.0 Detection Limits

3.1 Method detection limits (MDLs) are determined annually and results vary from element to element. Procedures for conducting MDL studies can be found in CT Laboratories Initial Method Performance and Reporting SOP (CL-2, rev. 5).

4.0 Scope and Application

4.1 Metals in solution can be readily analyzed by atomic emission using an Inductively Coupled Plasma (ICP) spectrometer. All matrices, excluding filtered groundwater samples and drinking waters with a turbidity less than 1 NTU, will require a digestion prior to analysis.

5.0 Method Summary

5.1 If necessary, prior to analysis, samples are digested using an approved method. See SOPs 6205B, 6225B, 6230B, M200.2, and M-soluble for further information on sample digestion.

5.2 This method describes multielement determinations using an iCAP 6000 Series ICP-OES which use an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Most Samples are liquids that are pumped through a nebulizer to produce a fine spray. The large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample is decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving measurement of the concentration of each element type in the original sample. Control of the spectrometer is provided by the PC based iTeva software (refer to the Thermo ICP-OES software manual). Samples are routinely analyzed using an internal standard solution of 50 mg/L Yttrium to eliminate certain matrix interference problems. Line switching is also used to extend the dynamic range of an element.

5.3 The data is exported to the LIMS system and reviewed by the analyst. Following analyst review, the data is given to a qualified reviewer for complete data review. After the data has been reviewed and it is determined that it is valid data, the reviewer sends the data to the "validated" mode in the LIMS system.

6.0 Definitions

6.1 Reagent Blank- A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

6.2 Calibration Standards - A series of known standard solutions, which shall include the reagent blank, used for calibration of the instrument within the measurable linear range. Calibration standards shall contain, in correct proportion, all reagents required by the method. Acceptance of the calibration requires a correlation coefficient (r) of 0.995 or

better. No samples shall be analyzed without acceptable calibration. For DOD-QSM data the low calibration standard shall be equal to or less than the MRL.

6.3 Calibration Verification Standard-Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. The ICV must be from a second source different than that of the calibration standards.

Note for method 200.7 that limits for ICV are tighter than those for the CCV (see section 16).

6.4 Low-Level Calibration Check Standard – If a single point calibration is used (for DOD-QSM data) then a low level standard shall be analyzed. The acceptance criterion is +/- 20% of the expected value.

6.5 CB (Calibration Blanks- Initial and Continuing) - A reagent blank solution, which is analyzed immediately following the ICV (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration, or possible analyte carry-over. Warning criteria include that the value be less than or equal to the three times the IDL for a given analyte for SW-846 work, less than 2x the MDL for DOD-QSM data, or less than ½ the MRL for ACOE work. Control criteria consist of the CB value being less than or equal to 2 times the MDL for a given analyte for SW-846 and is determined by the QAPP for DOD/ACOE work. If this range is exceeded, a new calibration will be necessary.

6.6 LCS (Laboratory Control Sample) - A mid-range standard, prepared from a source different from that used for calibration standards, which is carried through the entire preparation and analytical method. The LCS is used to verify the accuracy of the preparation method. A minimum of one LCS is prepared per batch and is analyzed at the beginning of an analytical batch.

6.7 MB (Method Blank) - A Reagent Blank (see 3.1) which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch and is analyzed at the beginning of an analytical batch. Blank recovery must be less than 2x the MDL for SW846, less than the MDL for SWDA samples, less than ½ the RL for DOD-QSM, and is determined by the QAPP for ACOE samples.

6.8 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every 20 samples of a given matrix per day for 6010B and once for every 10 samples of a given matrix per day for 200.7. For ACOE work only an MS is prepared and a duplicate sample is prepared rather than an MSD. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample.

6.9 Duplicate sample- A separate sample aliquot which is carried through the entire preparation and analytical procedure. A duplicate is prepared for every 20 samples for ACOE/LCG work and per batch for DOD-QSM (in replacement of an MSD).

6.10 Method Reporting Limit (MRL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near the reporting limit, or at a level

specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

6.11 Interelement Correction Factors (IEC) – These correction factors are determined by analyzing a concentration range of known interferents (Al, Ca, Co, Cr, Cu, Fe, Mg, Mn, Ni, V and Zn) and examining all other lines for a significant linear response. A line is considered to be significantly affected when the correlation coefficient for the interference is 0.99 or better and the correction factor multiplied by ten is greater than the MDL of the affected line. For interferents known to occur at high levels in environmental samples (Al, Ca, Cr, Cu, Fe, Mg and Zn) the interference will be considered to be significant when the correction factor multiplied by 100 is over the MDL of the affected line and the correlation coefficient in 0.99 or better. Interelement correction is used where applicable.

6.12 Linear Dynamic Range (LDR) – The upper limit of the linear dynamic range is established for each wavelength utilized by determining the signal responses from a minimum of three different concentration standards across the range. One of these will be near the upper limit of the range. The ranges used for the analysis of samples are judged by the analyst from the resulting data. The data and calculations are kept on file. The upper range limit is an observed signal no more than 10% below the level extrapolated from the lower standards. Determined analyte concentrations above the upper range limit are diluted and reanalyzed. New dynamic ranges are determined whenever there is a significant change in instrument response. For analytes that routinely approach the upper limit of the range, the range will be checked biannually. For analytes that are known interferents and exceed the dynamic range, the analyst will check that IEC's have been correctly applied. DOD-QSM requires that a LDR (or high level check standard) study be perform at least every six months.

Note: for ACOE/LCG work, analyte concentrations above the upper calibration limit are diluted and reanalyzed.

6.13 CS-A (Interelement Correction Standard-A): A standard containing the elements Al, Ca, Fe, and Mg at 500mg/L. This standard is analyzed when using method 200.7 or performing ACOE work following the ICV at the beginning of the run to determine that interelement correction factors are correctly compensating for interference from these elements on other analyte lines. The ICSA may be required to be run before the last CCV of the run for ACOE work. Check the QAPP to determine if this is necessary.

Note: For ACOE work, the ICSA should be within the absolute value of two times the MDL for all analytes except Al, Ca, Fe and Mg unless a different requirement is specified within the contract.

6.14 CS-AB (Interelement Correction Standard-AB): A standard containing the elements Al, Ca, Mg, and Fe at 500mg/L and all other elements at 500ug/L. This standard is analyzed following the ICV at the beginning of each run. It is analyzed to determine that the IEC are correctly preventing interference by these elements on the measurement of other analytes. The ICSAB may be required to be run before the last CCV of the run for ACOE work. Check the QAPP to determine if this is necessary

6.15 PDS (Post Digestion Spike): When a serial dilution or matrix spike falls outside of the acceptance limits a post digestion spike is used to determine if the sample digestion matrix is interfering with the analysis of the analyte. The sample is spiked at a level similar to that of the matrix spike.

Note: For ACOE/LCG work, a PDS will be conducted at a minimum rate of one per prep batch per unique matrix.

6.16 SD (Serial Dilution Analysis): A sample is diluted 5x with method blank solution and analyzed. The diluted result and the undiluted result should agree within a limit of precision defined by the program (SW846, CLP, 200.7) or client QAPP.

Note: For ACOE/LCG work, a SD will be conducted at a minimum rate of one per prep batch per unique matrix.

6.17 Batch- A batch consists of 20 samples of the same matrix analyzed on the same day or 20 samples of the same medium that have been prepared together.

6.18 IDL (Instrument Detection Limit); A series of blanks analyzed during initial setup or after significant changes, or as per DOD-QSM requirements. The limits established shall be $\leq$ MDL for any given element.

7.0 Interferences

7.1 Background emission and stray light are corrected using background correction. See ICP 6000 Series operator's manual for further instructions on background correction application.

7.2 Spectral overlaps are corrected for using interelement correction factors (IEC). When IEC are used, the interfering elements must be analyzed along with the elements of interest. The accuracy of IEC shall be verified daily by analyzing the ICSAB. All IEC factors shall be updated every six months or when an instrumentation change occurs; such as, changing a torch, nebulizer, injector, or plasma conditions.

7.3 Physical interferences such as viscosity are minimized by using an internal standard. Post digestion spike and serial dilutions help to determine if physical interferences are present.

7.4 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Chemical interferences are not normally seen during ICP analysis and are highly matrix dependent.

7.5 Memory interferences occur when a sample of high analyte concentration does not thoroughly rinse prior to the analysis of the next sample. This causes elevated readings for that analyte in the subsequent sample. Memory effects can be minimized by rinsing at least 60 seconds between samples.

8.0 Safety

8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

8.2 Insure that waste collection vessels contain enough room to accommodate all wastes that will be produced during the operation of the instrument.

9.0 Equipment and Supplies

9.1 ICP 6500, Cetac autosampler, computer, & network printer.

9.2 Argon (Airgas-liquid high purity or gaseous pre-purified grade or equivalent).

9.3 Class A volumetric flasks and pipettes (Chemglass or equivalent).

9.4 Disposable 15-mL polystyrene culture tubes.

9.5 100 uL pipette (Eppendorf or equivalent).

9.6 10-mL pipette (Eppendorf or equivalent).

10.0 Reagents and Materials

8.1 Reagents

8.2.1 Mixed and single element stock metals standards. See Section 9 and Appendix A, B, C and D for instructions on making the working standards.

8.2.2 Nitric acid, conc. (Fisher, Trace Metals grade or equivalent)
8.2.3 Hydrochloric acid, (Fisher, Trace Metals grade or equivalent)
8.2.4 Deionized water (Milli Q, > 10 mega ohm).

11.0 Sample Preservation and Storage

9.1 Samples must be preserved and analyzed within holding times stated in chart. Liquid samples are stored on shelves in the Laboratory warehouse and soil samples are stored in a walk-in refrigerator unit.

	Liquids	Solids
Preservative:	pH <2 with HNO_3	4^oC (+/-2)
Hold Time:	180 days	180 days

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 3. is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3 For the routine analysis of groundwater, wastewater, leachate, surface water, soil, sludge, TCLP/SPLP extracts following method 6010B:
Required QC following instrument calibration is as follows :

12.1.1 ICV (initial calibration verification); The ICV is prepared from an alternate source standard whose concentrations are within the linear working range of the instrument. The results of the ICV should agree within 10% of the expected value for a given analyte. The relative standard deviation (RSD) between the replicate integrations should be <5%. If results are outside of this range, corrective action must be taken before samples can be analyzed.

12.1.2 ICB (initial calibration blank); analyze the calibration blank. The results of the initial calibration blank must be < 3x IDL for a given analyte. If the average of the replicates is not < 3x IDL, terminate the analysis, correct the problem and recalibrate or appropriately qualify the data. If the blank is less than $1/10^{th}$ the concentration of the action level of interest and no sample is within ten percent of the action limit, analyses need not be rerun and re-calibration is not necessary before continuing with the analysis.

12.1.3 ICSAB (interference check solution); analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start troubleshooting .

12.1.4 LCS (laboratory control sample); analyze an alternate source reference sample. Control limits are +/- 20% of true value or in-house limits, whichever is more restrictive, or as specified in a client QAPP or the DOD-QSM manual.

12.1.5 MB (method blank); analyze a reagent blank. The method blank is a reagent blank that has been taken through the preparations steps along side the samples being analyzed. Control limits are the MDL. If the average of the replicates is not < MDL terminate the analysis, correct the problem and

recalibrate or appropriately qualify the data that falls within the MDL and twenty times the concentration of the analyte in the method blank.

12.1.5 CCV (continuing calibration verification); analyze a check standard after every ten samples and following the last sample in the run. This standard should be at a level approximately mid-scale. Control limits are +/- 10% of true value. If values fall outside this range, all samples back to the last acceptable ICV or CCV must be repeated.

12.1.6 CCB (continuing calibration blank); The results of the continuing calibration blank must be < 3x IDL for a given analyte. If the result falls outside this, reanalyze all samples back to the last acceptable CCB or qualify all sample <20 times the blank and greater than the MDL.

12.1.7 MS/MSD (matrix spike/matrix spike duplicate); for non-digested samples, prepare a bench spike in duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are +/- 25% of true value, and 20% RPD, or use calculated limits, whichever is tighter. See Sec. 18.0 for bench spike preparation. For digested samples, see "Predigestion Spike" chart in section 18.0. For digested samples, analyze the MS/MSD samples as they apply to each digestion set. Follow the above for control limits. For digested spikes with sample results greater than four times the digested spike level, prepare and analyze a PDS sample if the MS and/or MSD are outside the control limits. Prepare the PDS at a level approximately two times the sample level.

12.2 For SDWA analysis following method 200.7

Required QC following instrument calibration is as follows:

12.2.1 ICV: Referred to in 200.7 as LPC (laboratory performance check); analyze the mid-cal standard. Control limits are +/- 5% of true value. If values fall outside this range, recalibrate for the affected analytes.

12.2.2 ICB (initial calibration blank); analyze the calibration blank. The absolute value of the result should be below the MDL for the analyte(s) of interest. If the blank result falls outside this, evaluate the effect on the sample results and/or recalibrate for the affected analytes. Samples with results >10x the associated blank value need not be reanalyzed.

12.2.3 ICSA (interference check solution: interference only) analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe. This sample must be analyzed at the beginning of the analytical run before the ICSAB. Recovery for interfering analytes is +/- 20% true value. All other analytes need to be $\pm$ 2x the MDL. If recovery/result is outside the acceptable range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start trouble shooting.

12.2.4 ICSAB (interference check solution); analyze a solution containing 500 mg/L Al, Ca, Mg, Fe and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start trouble shooting.

12.2.5 LCS (laboratory control sample); analyze an alternate source reference sample per batch of 20 samples of the same matrix. Control limits are +/- 10% of true value, or manufacturer's limits, whichever is tighter.

12.2.6 MB (method blank); analyze a reagent blank per 20 samples of the same matrix. The method blank is a reagent blank that has been taken through the

preparations steps along side the samples being analyzed. If the average of the two replicates is not < MDL terminate the analysis, correct the problem and recalibrate or appropriately qualify the data that falls within the MDL and twenty times the concentration of the analyte in the method blank .

12.2.4 CCV: Referred to in 200.7 as LPC (laboratory performance check); analyze the mid-cal standard after every 10 samples. Control limits are +/- 10% of true value. If values fall outside this range, reanalyze all samples back to the last acceptable ICV or CCV.

12.2.5 CCB (continuing calibration blank); analyze the calibration blank immediately after each CCV. The value of the result should be below the MDL for the analyte(s)of interest. If the result falls outside this, evaluate the effect on the sample results. Sample results >10x the associated blank value need not be reanalyzed. Otherwise, reanalyze all samples back to the last acceptable CCB or qualify data that is >LOD and <10x the associated blank.

12.2.6 MS/MSD (matrix spike/matrix spike duplicate); for non-digested samples, prepare a bench spike in duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are +/- 25% of true value, and 20% RPD, or use calculated limits, whichever is tighter. See Sec. 18.0 for bench spike preparation. For digested samples, see "Predigestion Spike" chart in section 18.0. For digested samples, analyze the MS/MSD samples as they apply to each digestion set. Follow the above for control limits. For digested spikes with sample results greater than four times the digested spike level, prepare and analyze a PDS sample if the MS and/or MSD are outside the control limits. Prepare the PDS at a level approximately two times the sample level.

12.3 For the CLP-like level 4 analysis of groundwater, surface water, wastewater and soil

Note: A default of three replicate exposures per sample should be used for ACOE/LCG work unless specified differently in the QAPP.

Required QC following instrument calibration:

12.3.1 ICV (initial calibration verification): analyze the alternate source check standard immediately following calibration. Control limits +/- 10 % true value.

12.3.2 ICB (initial calibration blank): analyze the calibration blank. The absolute value of the result must be below the contract required detection limit (CRDL) or the limit stated within the QAPP for the project. The ICB results shall be < 2x the MDL for DOD-QSM data and <1/2 the RL for ACOE data. If a result falls outside this, recalibrate for the affected analytes.

12.3.3 CRDL (contract required detection limit standard) or MRL (Method Required Limit): analyze a standard at a level two times the contract-required detection limits (CRDL) or at the level stated within the QAPP for the project. Follow limits stated within the QAPP as there are no EPA specified control limits for this standard. This sample must be analyzed at the beginning and the end of the run.

12.3.4 ICSA (interference check solution: interference only) analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe. This sample must be analyzed at the beginning of the analytical run prior to the ICSAB. Refer to QAPP for acceptability criteria. For ACOE work, the default criteria is the absolute value of two times the MDL for all analytes except Al, Ca, Mg and Fe

which must have a recovery between 80-120%. Refer to QAPP to determine if the ICSA must also be analyzed at the end of the run.

12.3.5 ICSAB (interference check solution: interference plus analytes); analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe, and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points as a place to start troubleshooting. This sample must be analyzed at the beginning of the run. Refer to the QAPP to determine if the ICSAB must be analyzed at the end of the run.

12.3.6 Digested Sample set to include MB (S or W), LCS (S or W), MS, and DUP.

12.3.7 Serial Dilution: Analyze a x5 dilution of a sample from the digestion set. For sample results > 50x the MDL, the %RSD between the serial dilution result and the sample result must be $\leq$ 10.

12.3.8 Post digestion spike addition (bench spike): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within +/- 25% of the known value or as specified by the client QAPP. The spike addition should produce a minimum level of 10 times the instrumental detection limit. If the spike recovery falls outside the limits, a matrix effect should be suspected.

12.3.9 CCV (continuing calibration verification): Analyze a mid-level standard after every ten samples. The CRDL/MRL, ICSA, ICSAB and batch QC all count as samples. Control limits are +/- 10% of true value. If any result falls outside this, all samples back to the last acceptable ICV/CCV must be reanalyzed.

12.3.10 MB (method blank); analyze a reagent blank per 20 samples of the same matrix. The method blank is a reagent blank that has been taken through the preparations steps along side the samples being analyzed. If the average of the two replicates is not < MDL (< ½ the RL for DOD-QSM, or < project specified limits), terminate the analysis, correct the problem and recalibrate or appropriately qualify the data that falls within the MDL and twenty times the concentration of the analyte in the method blank.

12.3.11 CCB (continuing calibration blank); Analyze the calibration blank after each CCV (or every ten samples). Refer to the QAPP for CCB acceptance limits. The CCB results shall be < 2x the MDL for DOD-QSM data and <1/2 the RL for ACOE data. If any result falls outside these limits, all samples with results less 20 times the CCB must be reanalyzed back to the last acceptable CCB or appropriately qualified.

12.3.12 MS/DUP (matrix spike/matrix duplicate); for non-digested samples, prepare a bench spike and a duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are specified in client QAPP. or see "Predigestion Spikes"(Table 1). For digested samples, analyze the MS/DUP samples as they apply to each digestion set and follow Table 3 control limits.

12.4 New or unusual matrices: It is recommended that whenever a new or unusual sample matrix is encountered, a serial dilution and post digestion (bench) spike be performed prior to reporting results. These tests will ensure that neither positive nor negative interferences are affecting sample results.

Note: For ACOE work, a serial dilution and a post digestion spike will be performed at a rate of one per matrix with each prep batch.

12.4.1 Serial Dilution: If the analyte concentration is sufficiently high (minimally a factor of ten above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within +/- 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

12.4.2 Post digestion/bench spike addition (Table 2): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within +/- 25% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike recovery falls outside these limits, a matrix effect should be suspected..

13.0 Calibration and Standardization

13.1 The default calibration for TAL plus list (excluding Na and K) of metals for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called 'DOD calibration' which uses 12 mixed standards and a calibration blank.

13.2 The default calibration for the metals Sodium and Potassium for ACOE/ LCG, DOD-QSM and routine work is a multi-point calibration method called 'Sodium and Potassium' which uses 8 mixed standards and a calibration blank.

13.3 The default calibration for the metal Boron for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called 'Boron' which uses 5 standards and a calibration blank

13.4 The default calibration for the metal Lithium for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called 'Lithium' which uses 7 standards and a calibration blank. Refer to section 11.0 for further instructions on how to perform the calibration.

Note: See Appendix A for preparations of calibration standards and blank for the DOD calibration method calibration. See Appendix B for the preparation of the calibration standards and blank for the Sodium and Potassium method calibration. See Appendix C for the preparation of the calibration standards and blank for the Boron calibrations. See Appendix D for the preparation of the calibration standards and blank for the Lithium calibration. See Tables 4 (a,b,& c), 5, 6, and 7 for individual element calibration concentrations/ranges.

13.5 Calibration Blank: Into a 1 L. volumetric flask, add 750 mL of Milli-Q water and 10mL of conc. HNO3 and 10mL HCl. Mix, dilute to volume with Milli-Q H2O. Transfer to a clean 1 L. nalgene bottle. Prepare every 6 months or as needed.

13.6 Yttrium Internal std (Used for all methods) : Into a 2000mL volumetric flask, add 500mL of Milli-Q H2O, 10 ml 10,000 mg/L Yttrium standard dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 2L Nalgene bottle. Prepare every 6 months or as needed. Concentration = 50 mg/L Yttrium solution.

13.7 Initial/Continuing Calibration Verification (ICV/CCV): Into a 1000mL volumetric flask, add 500mL of Milli-Q water, 10 mL of conc. HNO3 and 10mL HCl. Add the following:

10 mL SPEX Certiprep Spike Sample Standard 1 or Equivalent
0.5mL Mo 1000mg/L
0.05ml Ag 1000mg/L
2 ml Interferents-A-SPEX Certiprep or equivalent

Dilute to volume with Milli-Q water, mix and transfer to a clean 1 L Nalgene bottle. Make new every 6 months or as needed.

Concentration	Analyte
50ug/L	Cd, Be
100ug/L	Ag
200ug/L	Cr
250ug/L	Cu
500ug/L	Co, Mn, Mo, Ni, Pb, Sb, V, Zn
2000ug/L	Ba, As, Tl, Se
5,000ug/L	Fe
10,000ug/L	Ca, Mg
12,000ug/L	Al

13.8 Interference Check Solution (ICSA): Into a 500 mL volumetric flask, add 300 mL of Milli-Q H2O, 5 mL of conc. HNO3 and 5mL conc. HCl. Add the following stock solutions in the volumes listed:

50 mL Spex Certiprep Interferents A or equivalent
15 ml Fe 10,000mg/L or equivalent

Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 500 mL Nalgene bottle. Prepare every 6 months or as needed.

Concentration	Analyte
500,000 ug/L	Al, Ca, Mg, Fe

13.9 Interference Check Solution (ICSAB): Into a 500 mL volumetric flask, add 300 mL of Milli-Q H2O, 5 mL of conc. HNO3 and 5 mL conc. HCl. Add the following stock solutions in the volumes listed:

50 mL Spex Certiprep Interferent A or equivalent
15 ml Spex Certiprep Fe 10,000 mg/L or equivalent
2.5 ml Spex Certiprep QC-21 or equivalent
0.25 ml Ultra Scientific Ag 1000 mg/L or equivalent
0.25 ml Ultra Scientific Ba 1000 mg/L or equivalent

Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 500 mL Nalgene bottle. Prepare every 6 months or as needed.

Concentration	Analyte
500,000 ug/L	Al, Ca, Mg, Fe
500 ug/L	Ag, As, Ba, Be Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, Zn

13.10 CRDL/MRL solution: Concentrations needed depend on the CRDL/MRL of a given contract.

14.0 Procedure

14.1 Instrument start-up procedure:

14.1.1 Open valve at argon tank, turn on chiller and instrument.. For best results, the instrument should be on and with an argon purge for at least 24 hours

14.1.2 Inspect pump tubing on instrument and on autosampler and change if necessary.

14.1.3 Fill DI rinse reservoir with DI water and preserve with HNO3 to1%.

14.1.4 Open up the ITeva software on the PC. Choose user, wait until instrument initializes.

14.1.5 Plasma startup and shutdown: Refer to the ICAP 6000 Series ICP-OES Spectrometer Operator manuals pages 11-1 thru 11-4. After plasma startup check the “Debug Wavelength Check” at the bottom of the ITeva control center. The absolute value of x and y #s should be less than 5, if not, stop and call Thermo service.

14.1.6 After a 30-minute warm-up period, check condition of nebulizer. Put pump tube into a 100mg/L solution Yttrium Std. With the lights off and after enough time has elapsed for the Yttrium standard to reach the plasma, a red cone should be noticeable in the center of the plasma. If the nebulizer is in good condition and the nebulizer gas flows are set properly, the red cone should project about 2mm beyond the coils. If not, check the settings, the pump tubes, and inspect the nebulizer under a microscope for any obstructions or breakage.

14.2 To create an autosampler sequence:

14.2.1 Refer to the ICAP 6000 Series ICP-OES Spectrometer Operator manuals pages 11-9 thru 11-11 to start an autosampler sequence.

14.2.2 Add all samples, LCS, Blanks, MS-MSD, etc. in order of program, agency or client request.

14.2.3 Print Autosampler Table: This will be used when preparing all samples and standards.

14.2.4 Using the printed autosampler table sheet, prepare all standards, QC samples, and samples in their designated positions in the autosampler. Prepare any bench spikes and place them in autosampler. Calibration standards, CCV, ICV, CCB, ICB and ICSAB all go into 50 ml vials in the designated S-# positions of the autosampler. All others are poured into 15 ml plastic vials into the designated areas within the 60 position racks.

14.3 Calibration and Analysis.

14.3.1 Once all calibration standards have been placed in the autosampler make sure the autosampler is initialized by clicking on the autosampler icon and the sequence is saved and then press the yellow arrow icon to start the calibration and prepare the remaining samples as the calibration is being carried out.

14.4 Instrument shutdown

14.4.1 If run will not be finished during work hours, program the instrument to shutdown at the end of the analytical run. When setting up on the sequence page click the “End Action (after all sequences) Box to “Shutdown Plasma”. This will shut down the plasma.

14.4.2 For manual shutdown go to the flame icon in the bottom right corner and click, and then select the plasma off icon. After the plasma is shutdown loosen the pump tubes and shut off the chiller.

15.0 Calculations

15.1 Sample Calculations:

Liquid Concentration (ug/L) = A x C

$$\textit{Solid Concentrations (mg/kg)} = \frac{A \times B \times C}{D \times E},$$

Where: A = instrument reading for sample (ug/L)
B = total volume of digestion (L)
C= dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary (fraction equivalent)

15.2 Spike Recovery Calculations:

$$\text{LCS Recovery (\%)} = \frac{\text{(Result obtained) x 100}}{\text{(Spike amount)}}$$

$$\textit{MS/MSDSpike Recovery (\%)} = \frac{\text{(Spiked sample conc. – Sample conc.) x 100}}{\text{(Spike amount)}}$$

$$\textit{\%RPD} = \frac{\text{(MS – MSD) x 100}}{\text{(MS + MSD)/2}},$$

Where: MS = Matrix spike concentration obtained
MSD = Matrix spike duplicate concentration obtained

16.0 Method Performance

16.1 Certified standard solutions, properly used instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

16.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (AR) and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the EPA methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated.

16.3 Proper instrument maintenance is another means to ensure adequate method performance.

16.3.1 Pump tubing and rollers: Ensure that the pump rollers turn freely. Inspect pump tubing daily and replace when it starts failing to retain its shape.

16.3.2 Drain line: Spray chamber drain line must flow unimpeded directly into waste jug. Make sure line is draining properly.

16.3.3 Spray Chamber: If the spray chamber becomes dirty, the sample waste may not drain properly. Remove and wash with hot soapy water, then rinse with DI H_2O.

16.3.4 Torch and O-rings: The O-rings surrounding the torch may need to be replaced if the plasma becomes unstable or internal standard emission counts fall off. See ICP6000 manual for technique. Torch needs to be cleaned occasionally with aqua regia followed by sonication. See sections 5 and 6 in the “ICAP 6000 Series ICP-OES Spectrometer Operating Manuals” for further assistance if needed.

17.0 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation.

17.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent

preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for QC Measures

18.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that excceedances are addressed. Acceptable data is then captured into the LIMS system (See SOP ICP6000 Data Capture for instructions on data capture).

18.2 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist (Table 2) for data review guidance.

18.3 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

18.4 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.

18.5 A paper hard copy of the data is then filed or archived. The package includes the checklist, the sequence run log, and a copy of the bench sheet (if applicable), the LIMS run log, and verification of calibration data.

19.0 Corrective Measures for Out-of-Control Data

19.1 When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities (Table 3.).

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

20.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.

20.1.1 The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

20.1.2 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

20.1.2.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

20.1.2.2 In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he or she instructs us to report the data, flag the data indicating what type of nonconformity has occurred.

20.1.2.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.

20.1.2.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

21.0 Waste Management

21.1 Samples are routinely held for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

22.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, EPA, SW-846, Method 6010B, rev. 2, December 1996.

17.2 Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, Method 200.7, rev. 4.4, 1991.

17.3 USEPA - Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0.

17.4 ICAP 6000 Series ICP-OES Spectrometer Operator's Manual, Thermo Electron (registration number 441506).

17.5 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, January, 2006.

17.6 Thermo Electron Corporation Training Manual iCAP 6000 Series.

17.7 Louisville Chemistry Guideline (LCG), US Army Corps of Engineers-Louisville District, June 2002.

17.8 Louisville DOD Quality Systems Manual Supplement (LS), US Army Corps of Engineers-Louisville District, March 2007.

Table1: Spike, LCS, & LFB Analysis- ICP
Pre-digestion Spikes

Element	Spike Amt. mL of	Spike Stock A, B, C	Stock Conc. mg/L	Final Vol. mL	Expected Conc. ug/L
Aluminum	1	**A**	200	50	4000
Antimony	1	**A**	50	50	1000
Arsenic	1	**A**	200	50	4000
Barium	1	**A**	200	50	4000
Beryllium	1	**A**	5	50	100
Cadmium	1	**A**	5	50	100
Calcium	0.5	**C**	20,000	50	200000
Chromium	1	**A**	20	50	400
Cobalt	1	**A**	50	50	1000
Copper	1	**A**	25	50	500
Iron	1	**A**	100	50	2000
Lead	1	**A**	50	50	1000
Manganese	1	**A**	50	50	1000
Magnesium	0.5	**C**	10,000	50	100000
Molybdenum	0.1	**B**	1000	50	2000
Nickel	1	**A**	50	50	1000
Selenium	1	**A**	200	50	4000
Silver	1	**A**	5	50	100
Thallium	1	**A**	200	50	4000
Vanadium	1	**A**	50	50	1000
Zinc	1	**A**	50	50	1000

.Spike Solutions

Supplier	Lot #/ std	Stock
SPEX Certiprep or equiv.	SPIKE 1-500	**A**
Molybdenum or equiv.	1000 mg/L	**B**
Custom Std	SPEX	**C**

Table 2: Bench Spike Analysis-ICP
Post Digestion/ Bench Spikes

Element	Spike Amt. ML of	Spike Soln.	Stock Conc. mg/L	Final Vol. mL	Expected Conc. ug/L
Aluminum	0.2	**A**	200	10	4000
Antimony	0.2	**A**	50	10	1000
Arsenic	0.2	**A**	200	10	4000
Barium	0.2	**A**	200	10	4000
Beryllium	0.2	**A**	5	10	100
Cadmium	0.2	**A**	5	10	100
Calcium	0.2	**B**	20,000	10	400000
Chromium	0.2	**A**	20	10	400
Cobalt	0.2	**A**	50	10	1000
Copper	0.2	**A**	25	10	500
Iron	0.2	**A**	100	10	2000
Lead	0.2	**A**	50	10	1000
Manganese	0.2	**A**	50	10	1000
Magnesium	0.2	**B**	10,000	10	200000
Molybdenum	0.02	**C***	1000	10	2000
Nickel	0.2	**A**	50	10	1000
Selenium	0.2	**A**	200	10	4000
Silver	0.2	**A**	5	10	10
Thallium	0.2	**A**	200	10	4000
Vanadium	0.2	**A**	50	10	1000
Zinc	0.2	**A**	50	10	1000

Standard Source	
Supplier	
SPEX	**A**
SPEX Custom Std	**B**
Molybdenum-1,000 mg/L Std	**C***

C*: substock of 1,000 mg/L std. (1:10 dilution.)

Table 3: Summary of Quality Control Requirements

QC Type	Frequency	Conc. Level	*Acceptance Criteria*	*Corrective Action*
ICV	1 per calibration	Mid.Cal Range	SDWA: 95-105% SW846:90-110% or Project Specific	Terminate run. Correct the problem before proceeding
ICB	Immediately after the ICV	<MDL	SW846: < 3x IDL SDWA: < MDL DOD-QSM: <1/2 MRL or Project Specific	Terminate analysis and correct the problem before proceeding.
MB	1 per batch of 20 samples	<MDL	SDWA: < MDL SW846: < 2x MDL QSM-DOD: <1/2 MRL or Project Specific	Access data and reanalyze/reprepare the MB and affected data or flag "B" analyte detected in Method Blank when insufficient sample remains
LCS	1 per batch of 20 samples	mid cal. Range	In-house limits or, default 80-120% SDWA; 90-110% or Project Specific	Terminate analysis: correct problem before proceeding.
CCV	1 after every 10th sample	mid cal range	SW846: 90-110% SDWA: 90-110% CLP:90-110%	Recalibrate and reanalyze all samples back to the last acceptable CCV or ICV
CCB	Immediately following each CCV	<MDL	SW846: < 3x IDL SDWA: < MDL DOD-QSM: < 2x MDL or Project Specific	Reanalyze all samples back to the last acceptable CCB or flag "B" analytes detected
ICSA	Immediately After LCS (&before final CCV if required by QAPP)	500mg/L Al, CA, Mg 200mg/L Fe (non –ACOE) or 500mg/L Fe (ACOE)	80-120% for Interference Elements ABS of analytes not included must be < 2X MRL or Project Specific	Terminate analysis, correct problem & reanalyze all samples back to last good ICSA/ICSAB
ICSAB	Immediately After ICSA (& before final CCV if required by QAPP)	500mg/L Al, CA, Mg 200mg/L Fe (non-ACOE) or 500 mg/L Fe (ACOE) Other elements 500ug/L	80-120% for All Elements	Terminate analysis, correct problem & reanalyze all samples back to last good ICSA/ICSAB
MS-MSD	5% (1 in 20) of samples per batch per matrix	See attached spike chart	In-house limits, Project Specific, or, default of 75-125 when spike level is >25% of original analyte level	Perform PDS

Serial Dilution Analysis	1 per digestion batch per matrix	5 fold dilution of chosen sample	RPD within 10% of value of diluted and undiluted sample, but only if sample conc. >/= 50 X's LOD	Flag data only if sample conc. Is within range (>50X's LOD)
Post Digestion Spike (PDS)	Upon failure of MS and/or 1 per batch for ACOE work	Same level as MS	85-115%	Flag: Matrix interference or Method of Std. Additions

Table 4a. TAL list Concentrations/Ranges for ICP ug/L

Element, Wavelength and Order	Blank		CalibStd=0.25		CalibStd=0.5		CalibStd=1		CalibStd=5		CalibStd=10		C
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?
Ag 328.068 {103}	☒	0	☐		☐		☐		☒	5	☒	10	☒
Ag 338.289 {100}	☒		☐		☐		☐		☐		☐		☐
Al 167.079 {502}	☒	0	☐		☐		☐		☐		☒	10	☒
Al 309.271 {109}	☒		☐		☐		☐		☐		☐		☐
Al 396.152 { 85}	☒		☐		☐		☐		☐		☐		☐
As 189.042 {479}	☒	0	☐		☐		☐		☐		☐	10	☐
As 193.759 {474}	☒		☐		☐		☐		☐		☒		☐
Ba 455.403 { 74}	☒	0	☐		☐		☒	1	☒	5	☒	10	☒
Ba 493.409 { 68}	☒		☐		☐		☐		☐		☐		☐
Be 234.861 {144}	☒	0	☐	0.25	☐	0.5	☐	1	☐	5	☐	10	☐
Be 313.042 {108}	☒		☒		☒		☒		☒		☒		☒
Ca 317.933 {106}	☒	0	☐		☐		☐		☐		☐	10	☐
Ca 393.366 { 86}	☒		☐		☐		☐		☐		☒		☒
Ca 396.847 { 85}	☒		☐		☐		☐		☐		☐		☐
Cd 226.502 {449}	☒	0	☒	0.25	☒	0.5	☒	1	☒	5	☐		☒
Cd 228.802 {447}	☒		☐		☐		☐		☐		☐		☐
Co 228.616 {447}	☒	0	☐		☐		☒	1	☒	5	☒	10	☐
Co 238.892 {141}	☒		☐		☐		☐		☐		☐		☐
Cr 205.552 {464}	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
Cr 267.716 {126}	☒		☐		☐		☒		☒		☒		☒
Cu 224.700 {450}	☒	0	☐		☐		☐		☐	5	☐		☒
Cu 327.396 {103}	☒		☐		☐		☐		☒		☐		☒
Fe 234.349 {144}	☒	0	☐		☐		☐		☐		☐	10	☐
Fe 239.562 {141}	☒		☐		☐		☐		☐		☐		☐
Fe 259.940 {130}	☒		☐		☐		☐		☐		☒		☐
Mg 279.079 {121}	☒	0	☐		☐		☐		☐		☐	10	☐
Mg 279.553 {121}	☒		☐		☐		☐		☐		☒		☐
Mg 280.270 {120}	☒		☐		☐		☐		☐		☐		☐
Mn 257.610 {131}	☒	0	☐		☐		☐		☒	5	☒	10	☒
Mn 259.373 {130}	☒		☐		☐		☐		☐		☐		☐
Mo 202.030 {467}	☒	0	☐		☐		☒	1	☐		☒	10	☒
Mo 204.598 {465}	☒		☐		☐		☐		☐		☐		☐
Ni 221.647 {452}	☒	0	☐		☐		☐	1	☐		☐	10	☐
Ni 231.604 {445}	☒		☐		☐		☒		☐		☒		☒
Pb 216.999 {455}	☒	0	☐		☐		☐	1	☐		☐	10	☐
Pb 220.353 {453}	☒		☐		☐		☒		☐		☒		☒
Sb 206.833 {463}	☒	0	☐		☐		☐		☐	5	☐	10	☐
Sb 217.581 {455}	☒		☐		☐		☐		☒		☒		☒
Se 196.090 {472}	☒	0	☐		☐		☒	1	☐		☒	10	☐
Se 206.279 {463}	☒		☐		☐		☐		☐		☐		☐
Tl 190.856 {477}	☒	0	☐		☐		☐		☒	5	☒	10	☒
Tl 351.924 { 98}	☒		☐		☐		☐		☐		☐		☐
V 290.882 {116}	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
V 292.402 {115}	☒		☐		☐		☒		☒		☒		☒
Y 360.073 { 94}*	☒	0	☐		☐		☐		☐		☐		☐
Zn 206.200 {463}	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
Zn 213.856 {458}	☒		☐		☐		☒		☒		☒		☒

Table 4b. TAL list Concentrations/Ranges for ICP ug/L cont.

Element, Wavelength and Order	Calibration Standards													
	alibStd=20		CalibStd=50		CalibStd=100		CalibStd=500		CalibStd=1000		CalibStd=5000		CalibStd=10000	
	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?
Ag 328.068 (103)	20	X	50	X	100		500		1000					
Ag 338.289 (100)														
Al 167.079 (502)	20	X	50	X	100	X	500	X	1000	X	5000		10000	
Al 309.271 (109)														
Al 396.152 (85)														
As 189.042 (479)			50		100		500	X	1000	X	5000	X	10000	
As 193.759 (474)														
Ba 455.403 (74)	20	X	50		100	X	500	X	1000	X	5000		10000	
Ba 493.409 (68)														
Be 234.861 (144)	20		50		100		500	X	1000	X	5000	X	10000	
Be 313.042 (108)														
Ca 317.933 (106)	20				100		500		1000	X	5000	X	10000	
Ca 393.366 (86)														
Ca 396.847 (85)														
Cd 226.502 (449)	20	X	50	X	100	X	500	X	1000		5000		10000	
Cd 228.802 (447)														
Co 228.616 (447)		X	50	X	100	X	500	X	1000	X	5000		10000	
Co 238.892 (141)														
Cr 205.552 (464)	20		50		100		500		1000	X	5000	X	10000	
Cr 267.716 (126)		X		X		X		X						
Cu 224.700 (450)	20	X	50	X	100	X	500	X	1000	X	5000			
Cu 327.396 (103)		X		X		X								
Fe 234.349 (144)			50		100		500		1000		5000	X	10000	
Fe 239.562 (141)														
Fe 259.940 (130)		X		X		X		X		X				
Mg 279.079 (121)			50		100		500		1000	X	5000	X	10000	
Mg 279.553 (121)		X		X		X		X						
Mg 280.270 (120)														
Mn 257.610 (131)	20	X	50	X	100	X	500	X	1000	X	5000		10000	
Mn 259.373 (130)														
Mo 202.030 (467)	20	X	50	X	100	X	500	X	1000		5000		10000	
Mo 204.598 (465)														
Ni 221.647 (452)	20		50		100		500		1000	X	5000	X	10000	
Ni 231.604 (445)		X		X		X		X						
Pb 216.999 (455)	20		50		100		500	X	1000	X	5000	X	10000	
Pb 220.353 (453)		X		X		X								
Sb 206.833 (463)	20				100		500	X	1000	X	5000	X	10000	
Sb 217.581 (455)														
Se 196.090 (472)		X	50	X	100	X	500	X	1000	X	5000		10000	
Se 206.279 (463)														
Tl 190.856 (477)	20	X	50	X	100	X	500	X	1000	X	5000	X	10000	
Tl 351.924 (96)														
V 290.882 (116)	20		50		100	X	500	X	1000	X	5000	X	10000	
V 292.402 (115)		X		X		X								
Y 360.073 (94)*														
Zn 206.200 (463)	20		50		100		500		1000	X	5000	X	10000	
Zn 213.856 (458)		X		X		X		X		X				

Table 4c. TAL list Concentrations/Ranges for ICP ug/L cont.

Element, Wavelength and Order	CalibStd-100k		CalibStd-100000		CalibStd-500000		CalibStd-1000k	
	?	Conc	?	Conc	?	Conc	?	Conc
Ag 328.068 {103}	☐		☐		☐		☐	
Ag 338.289 {100}	☐		☐		☐		☐	
Al 167.079 {502}	☐		☐	100000	☐	500000	☐	1e+006
Al 309.271 {109}	☐		☒		☐		☐	
Al 396.152 { 85}	☐		☒		☒		☒	
As 189.042 {479}	☐		☐		☐		☐	
As 193.759 {474}	☐		☐		☐		☐	
Ba 455.403 { 74}	☐		☐		☐		☐	
Ba 493.409 { 68}	☐		☐		☐		☐	
Be 234.861 {144}	☐		☐		☐		☐	
Be 313.042 {108}	☐		☐		☐		☐	
Ca 317.933 {106}	☐		☒	100000	☒	500000	☒	1e+006
Ca 393.366 { 86}	☐		☐		☐		☐	
Ca 396.847 { 85}	☐		☒		☐		☐	
Cd 226.502 {449}	☐		☐		☐		☐	
Cd 228.802 {447}	☐		☐		☐		☐	
Co 228.616 {447}	☐		☐		☐		☐	
Co 238.892 {141}	☐		☐		☐		☐	
Cr 205.552 {464}	☒	100000	☐		☐		☐	
Cr 267.716 {126}	☐		☐		☐		☐	
Cu 224.700 {450}	☒	100000	☐		☐		☐	
Cu 327.396 {103}	☐		☐		☐		☐	
Fe 234.349 {144}	☐		☒	100000	☒	500000	☒	1e+006
Fe 239.562 {141}	☐		☐		☐		☐	
Fe 259.940 {130}	☐		☐		☐		☐	
Mg 279.079 {121}	☐		☒	100000	☒	500000	☒	1e+006
Mg 279.553 {121}	☐		☐		☐		☐	
Mg 280.270 {120}	☐		☐		☐		☐	
Mn 257.610 {131}	☐	100000	☐		☐		☐	
Mn 259.373 {130}	☒		☐		☐		☐	
Mo 202.030 {467}	☐		☐		☐		☐	
Mo 204.598 {465}	☐		☐		☐		☐	
Ni 221.647 {452}	☐		☐		☐		☐	
Ni 231.604 {445}	☐		☐		☐		☐	
Pb 216.999 {455}	☒	100000	☐		☐		☐	
Pb 220.353 {453}	☐		☐		☐		☐	
Sb 206.833 {463}	☐		☐		☐		☐	
Sb 217.581 {455}	☐		☐		☐		☐	
Se 196.090 {472}	☐		☐		☐		☐	
Se 206.279 {463}	☐		☐		☐		☐	
Tl 190.856 {477}	☐		☐		☐		☐	
Tl 351.924 { 96}	☐		☐		☐		☐	
V 290.882 {116}	☐		☐		☐		☐	
V 292.402 {115}	☐		☐		☐		☐	
Y 360.073 { 94}*	☐		☐		☐		☐	
Zn 206.200 {463}	☒	100000	☐		☐		☐	
Zn 213.856 {458}	☐		☐		☐		☐	

Note: These metals are calibrated using a blank and minimum of three standards. It is not allowable to remove any mid levels to obtain an acceptable calibration; all points must used. Multi-level calibrations must be sequential.

Appendix A.

Element specific standard prep for multipoint calibration of ICP.

A1 Calibration Standard 0.25 (Be, & Cd): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.25 ul of SPEX Quality Control Standard 7 and 0.25 uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-

Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.25 ug/L.

A2 Calibration Standard 0.50 (Be & Cd): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 50uL of SPEX Quality Control Standard 7 and 50uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.50 ug/L.

A3 Calibration Standard 1.0 (Ba, Be, Cd, Co, Cr, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1 ml of conc. HNO3 and 1mL conc. HCl. Add 1.0 uL of SPEX Quality Control Standard 7 and 1.0 uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 ug/L

A4 Calibration Standard 5.0 (Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5 uL of SPEX Quality Control Standard 7 and 5 uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5 ug/L

A5 Calibration Standard 10.0 (Ag, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 10 uL of SPEX Quality Control Standard 7 and 10 uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 ug/L

A6 Calibration Standard 20 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe Mn, Mo, Ni, Pb, Sb, Tl, & V): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 20uL of SPEX Quality Control Standard 7 and 20uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 20 ug/L

A7 Calibration Standard 50 (Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 50uL of SPEX Quality Control Standard 7 and 50uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 ug/L

A8 Calibration Standard 100 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 100 uL of SPEX Quality Control Standard 7 and 100 uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 ug/L

A9 Calibration Standard 500 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 500uL of SPEX Quality Control Standard 7 and 500uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L

A10 Calibration Standard 1000 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1000uL of SPEX Quality Control Standard 7 and 1000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L

A11 Calibration Standard 5000 (Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5000uL of SPEX Quality Control Standard 7 and 5000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5000 ug/L

A12 Calibration Standard 10000 (Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 10000uL of SPEX Quality Control Standard 7 and 10000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10000 ug/L

A13 Calibration Standard 100000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 5mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 1mL of 10,000 mg/L Al, 1mL of 10,000 mg/L Ca, 1mL of 10,000 mg/L Fe and 1mL of 10,000 mg/L Mg . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100,000 ug/L Al, Ca, Fe, Mn, and Mg.

A14 Calibration Standard 100K (Cr, Cu, Co, Ni, Mn, & Pb): Into a 100mL volumetric flask, add 5mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl and add 10 mls of Cr 1000 mg/L, 10 mls Cu 1000 mg/L, 10 mls Pb 1000 mg/L, 10 mls Co 1000 mg/L,10 mls Ni 1000 mg/L and 10 ml of Mn 1000 mg/L and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration:100,000 ug/L Co, Cr, Cu, Ni, Mn, and Pb.

A15 Calibration Standard 500000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 5mL of 10,000 mg/L Al, 5mL of 10,000 mg/L Ca, 5mL of 10,000 mg/L Fe and 5mL of 10,000 mg/L Mg. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500,000 ug/L Al, Ca, Fe and Mg

A16 Calibration Standard 1000000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 10mL of 10,000 mg/L Al, 10mL of 10,000 mg/L Ca, 10mL of 10,000 mg/L Fe and 10mL of 10,000 mg/L Mg. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1,000,000ug/L Al, Ca, Fe and Mg

A17 Calibration Blank: Into a 1 L. volumetric flask, add 750 mL of Milli-Q water and 10 mL of conc. HNO3 and 10mL HCl. Mix, dilute to volume with Milli-Q H2O. Transfer to a clean 1 L. nalgene bottle. Prepare every 6 months or as needed.

A18 ICV/CCV: Into a 1000mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 10mL of conc. HCl. Add 10.0mL Spex Certiprep Spike Sample Standard 1 or equivalent and 2.0mL Interference A or equivalent, 0.5 mls of 1000 ug/L Mo and 0.05 mls 1000 ug/L Ag . Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L

A19 ICSAB: Into a 100mL volumetric flask, add 500mL Milli-Q water, 5mL of conc. HNO3 and 5mL of conc. HCl. Add 50 ml of SPEX Interferents A or equivalent, 15.0mLs of 10,000 mg/L Fe, 2.5mL Spex Certiprep QC 7 and 2.5 ml Spex Certiprep-QC 21 or equivalent. Dilute to volume with Milli-Q water and mix by inverting several times.

Table 5. Sodium and Potassium Concentrations/Ranges for ICP (mg/L)

Element, Wavelength and Order	Calibration Standards																	
	Blank		CalibStd-0.5		CalibStd-1.0		CalibStd-5.0		CalibStd-10.0		CalibStd-50.0		CalibStd-100		CalibStd-200		CalibStd-500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
K 766.490 (44)	☒	0	☒	0.5	☒	1	☒	5	☒	10	☒	50	☒	100	☒	200	☐	
Na 330.237 (102)	☒	0	☐	0.5	☐	1	☐	5	☐	10	☒	50	☒	100	☒	200	☐	
Na 589.592 (57)	☒		☒		☒		☒		☒		☒		☐		☐		☐	
Al 309.271 (109)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Ca 317.933 (106)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Fe 259.940 (130)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Mg 279.079 (121)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Y 360.073 (94)*	☒	0	☐		☐		☐		☐		☐		☐		☐		☐	

Note: These metals are calibrated using a blank and minimum of three standards. It is not allowable to remove any mid levels to obtain an acceptable calibration; all points must be used. Multi-level calibrations must be sequential.

Appendix B

Standard Prep for Sodium and Potassium analysis.

B1 Calibration Standard 0.5: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 2mL of conc. HNO3 and 2mL conc. HCl. Add 0.05mL Sodium 1000mg/L and 0.05mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.5 mg/L Na, K.

B2 Calibration Standard 1.0: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.1mL Sodium 1000mg/L and 0.1mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer

to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 mg/L Na, K.

B3. Calibration Standard 5.0: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.5mL Sodium 1000mg/L and 0.5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5 mg/L Na, K.

B4. Calibration Standard 10: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1mL Sodium 1000mg/L and 5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 mg/L Na, K.

B5. Calibration Standard 50: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5mL Sodium 1000mg/L and 5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 mg/L Na, K.

B6. Calibration Standard 100: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 10mL Sodium 1000mg/L and 10mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L Na, K.

B7. Calibration Standard 200: Into a 100mL volumetric flask, add H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 50mL Sodium 1000mg/L and 50mL Potassium 1000mg/L. Mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Na, K.

B8. Calibration Standard 500 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

B9. ICV/CCV: Into a 100mL volumetric flask, add 50mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 1.0mL 10,000mg/L Na and 1.0mL 10,000 K. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L Na an K.

B8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 2.0mL 10,000mg/L Na and 2.0mL 10,000 K. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 200mg/L Na and K, 500,000ug/L Al, Ca, Fe and Mg.

Table 6. Boron and Silicon Concentrations/Ranges for ICP (ug/L)

Element, Wavelength and Order	Calibration Standards													
	Blank		CalibStd=50		CalibStd=50 &10		CalibStd=500 &1		CalibStd=10000&2		CalibStd=50000&1		CalibStd=500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
B 249.773 {135}	☒	0	☒	50	☒	100	☒	1000	☒	2000	☒	10000	☐	
Si 251.611 {134}	☒	0	☐		☒	50	☒	500	☒	1000	☒	5000	☐	
Al 308.215 {109}	☒	0	☐		☐		☐		☐		☐		☒	500000
Ca 317.933 {106}	☒	0	☐		☐		☐		☐		☐		☒	500000
Fe 234.349 {144}	☒	0	☐		☐		☐		☐		☐		☒	500000
Mg 279.079 {121}	☒	0	☐		☐		☐		☐		☐		☒	500000
Y 360.073 {94}*	☒	0	☐		☐		☐		☐		☐		☐	

Note: Boron and Silicon are calibrated using a blank and a minimum three standards. It is not allowable to remove any mid levels to obtain an acceptable calibration; all points must be used. Multi-level calibrations must be sequential.

Appendix C

Standard Prep for Boron and Silicon Analysis

C1. Calibration Standard 50 ug/L B: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.05 ml Spex Certiprep QC std. 7 or equivalent. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed.Concentration: 50 ug/L B

C2. Calibration Standard 50 ug/L Si & 100 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.1 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 ug/L Si & 100 ug/L B

C3. Calibration Standard 500 ug/L Si & 1000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Si &1000 ug/L B.

C4. Calibration Standard 1000 ug/L Si & 2000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration:1000 ug/L Si &5000 ug/L B.

C5. Calibration Standard 5000 ug/L Si & 10000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5000 ug/L Si &10000 ug/L B.

C6. Calibration Standard 500 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

C7. ICV/CCV: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q water, 1 mL of conc HNO3 and 1mL conc. HCl. Add 0.1mL of 1000mg/L Boron and 0.1mL of 1000mg/L Silicon (alternate sources from calibration source). Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L Boron and Silicon.

C8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 0.05 ml 1000 mg/L B and 0.5 ml 1000 mg /L Si. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L B and Si, 500,000ug/L Al, Ca, Fe and Mg.

Table 7. Lithium, Tin, Strontium, Titanium, and Tungsten Concentrations/Ranges for ICP ug/L

Element, Wavelength and Order	Calibration Standards															
	Blank		1		10		100		1000		10000		500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
Li 670.784 (50)	☒	0	☒	1	☒	10	☒	200	☒	1000	☒	10000	☐	
Sn 189.989 (477)	☒	0	☐		☐	10	☐	100	☒	1000	☒	10000	☐	
Sn 189.989 (478)	☒		☐		☐		☐		☐		☐		☐	
Sr 407.771 (83)	☒	0	☒	1	☐	10	☒	100	☒	1000	☐	10000	☐	
Sr 421.552 (80)	☒		☐		☐		☐		☐		☐		☐	
Ti 334.941 (101)	☒	0	☐	1	☐	10	☒	100	☒	1000	☒	10000	☐	
Ti 337.280 (100)	☒		☒		☐		☐		☐		☐		☐	
W 224.875 (450)	☒	0	☐		☐	10	☐	100	☒	1000	☐	10000	☐	
W 239.709 (141)	☒		☐		☐		☐		☒		☒		☐	
Al 309.271 (109)	☒	0	☐		☐		☐		☐		☐		☒	500000
Ca 317.933 (106)	☒	0	☐		☐		☐		☐		☐		☒	500000
Fe 259.940 (130)	☒	0	☐		☐		☐		☐		☐		☒	500000
Mg 279.553 (121)	☒	0	☐		☐		☐		☐		☐		☒	500000
Y 360.073 (94)*	☒	0												

Appendix D

Standard prep for Lithium, Tin, Titanium, Strontium, and Tungsten

D1. Calibration Standard 1: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 0.01 ml Spex Certiprep QC std. 21 or equivalent and .001 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 ug/L

D2. Calibration Standard 10: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 0.1 ml Spex Certiprep QC std. 21 or equivalent and .01 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 ug/L Li, Sr, Sn, Ti, and W.

D3. Calibration Standard 100: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 21 or equivalent and 0.1 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting

several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 ug/L Li, Sr, Sn, Ti, and W.

D4. Calibration Standard 1000: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 10.0 ml Spex Certiprep QC std. 21 or equivalent and 1.0 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L Li, Sr, Sn, Ti, and W.

D5. Calibration Standard 10000: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 100.0 ml Spex Certiprep QC std. 21 or equivalent and 10.0 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10000 ug/L Li, Sr, Sn, Ti, and W.

D6. Calibration Standard 500000 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

D7. ICV/CCV: Into a 1000mL volumetric flask, add 50mL of Milli-Q water, 10 ml of conc. HNO3 and 10mL conc. HCl. Add 0.5mL of 1000mg/L Li, 0.5mL of 1000ug/L Sn, 0.5mL of 1000ug/L Sr, 0.5 ml of 1000mg/L Silicon, 0.5 ml 1000mg/L Tungsten and 0.5 ml of 1000mg/L Titanium. (all alternate sources from calibration source). Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Li, Sr, Sn, Ti, and W.

D8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 0.05 ml of 1000 mg/L Sr, 0.05 ml 1000 mg /L of Li, 0.05 ml 1000 mg /L of Sn, 0.05 ml 1000 mg /L of Ti, 0.05 ml 1000 mg /L of W. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Li, Sr, Sn, Ti, and W and 500,000ug/L Al, Ca, Fe and Mg.

Note: For DOD-QSM data the lowest level (Calib. Level # 1) on a multi point curve is prepared at concentrations equal to or less than the MRL for any given project and these levels are subject to change.

Table 8: Data Review Checklist

ICP6500 OES Data Review Checklist	Analysis Date:		Data File:		Date review:
Cal Std ID: LIMS # :	Analyst:		Reviewer:		Approved? Yes No
Calibration Parameters - 6010 200.7	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV 90-110% 95-105%					
3) ICB ABS 3X DL ABS DL					
4) ICSAB 80-120% True Value					
5) ICSA +/- 2X RL or LOD					
6) MRL - (2X RL or LOD) 70-130%					
7) CCV1/CCB1-90-110% / 3X DL ABS DL					
8) CCV2/CCB2					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
12) CCV6/CCB6					
13) CCV7/CCB7					
14) CCV8/CCB8					
15) CCV9/CCB9					
16) CCV10/CCB10					
Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	**Comments:**
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits; attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 75-125% sample#_____					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 75-125% sample#_____					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits; attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					

CT Laboratories

Title: **Mercury Cold Vapor Atomic Absorption**

SOP Number: **6120B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

This method is appropriate for measuring mercury concentrations in groundwater, wastewater, drinking water, TCLP extracts, soils, sediments, and sludge-type materials.

2.0 METHOD SUMMARY

2.1 Prior to analysis, the samples must be prepared according to the procedures discussed in this SOP.

2.2 This is a cold-vapor atomic absorption technique, based on the absorption of radiation at 254-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3.0 DEFINITIONS

3.1 Reagent Blank - A solution of deionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.2 Calibration Standards - A series of known standard solutions used for calibration of the instrument within the measurable linear range. Calibration standards shall contain, in correct proportion, all reagents required by the method. A total of 5 calibration points are used for Mercury calibration. Acceptance of the calibration requires a correlation coefficient of 0.995 or better. No samples shall be analyzed without acceptable calibration.

3.3 Calibration Verification Standards-Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. The ICV must be from a second source different than that of the calibration standards, while the CCV may be from the same source as the calibration standards. Note that limits for ICV are tighter than those for CCV (see section 16).

3.4 CB (Calibration Blanks- Initial and Continuing) - A reagent blank solution, which is analyzed immediately following the calibration standards (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration or possible analyte carry-over. Control criteria consist of the highest of the following: the absolute value being less than or equal to the MDL for a given analyte for routine work, <2x the MDL for DOD-QSM, or <½ the MRL for ACOE work. . If these ranges are exceeded, correct the problem and reanalyze affected data. A new calibration may be necessary to correct the problem.

3.5 LCS (Laboratory Control Sample)- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the digestion and is analyzed at the beginning of the analytical batch.

3.6 MB (Method Blank) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch. Method blank value should be lower than the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit of five percent of the measure concentration in the sample. The MB results shall also be < ½ the MRL for DOD-QSM/ACOE data.

3.7 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every 20 samples per matrix of routine samples or for ACOE a DUP/MS pair is prepared for every 20 samples of a given matrix per day. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD and may be used as acceptance criteria for duplicate analysis.

3.8 Method Reporting Limit (MRL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near but below the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

3.9 Duplicate (DUP)- A separate aliquot of sample which has been carried through the entire preparation and analytical procedure the same as the original sample. One duplicate per batch is prepared for ACOE work.

3.10 SD (Serial Dilution Analysis): A sample is diluted 1:5 with method blank solution and analyzed. The diluted result and the undiluted result should agree within a limit of precision defined by the program (SW846, CLP, 200.7) or client QAPP. **For ACOE/QSM work, a SD will be conducted at a minimum rate of one per prep batch per unique matrix.**

4.0 HEALTH AND SAFETY

4.1 Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure shall utilize appropriate laboratory safety systems.

5.0 CAUTIONS

5.1 Mercury is a toxic substance and care should be taken to avoid contact with it. This includes wearing gloves and using ventilation devices when working with Mercury.

5.2 This method allows for detection of small quantities of Mercury. All potential sources of Mercury contamination should be avoided. This would include sources of Mercury present in other lab areas.

6.0 INTERFERENCES

6.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

6.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

6.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

6.4 Certain volatile organic materials that absorb at this wavelength mayalso cause interference. A preliminary run without reagents should determine if this type of interference is present.

7.0 PERSONNEL QUALIFICATIONS

7.1 Personnel operating the CVAA shall have background knowledge of the scientific principles used during this application. All operators shall perform an initial demonstration of capability (IDC) prior to analyzing any samples. It is preferable for the operator to have at least two semesters of college chemistry.

8.0 APPARATUS AND MATERIALS

8.1 APPARATUS & MATERIALS

8.1.1 Cetac M-6000A Mercury Analyzer with ASX-500 autosampler.

8.1.2 Argon gas, HP grade.

8.1.3 50 mL disposable centrifuge tubes and caps. (Fisher p/n 05-539-9 or equivalent)

8.1.4 25 mL Class A volumetric pipettes

8.1.5 100 mL volumetric flasks

8.1.6 25 mL glass Class A, TD, graduated cylinders

8.1.7 1, 2, 3, 4, 5 mL Class A volumetric pipettes.

8.1.8 Eppendorf pipette, 0.100 to 1.000 mL range.

8.1.9 Environmental Express Hot Blocks set at 90-95 ^{0}C.

8.2 REAGENTS

8.2.1 Sulfuric acid, H_2SO_4, concentrated: Trace metal grade (Fisher p/n A300C-212)

8.2.2 Nitric acid, HNO_3, concentrated: Trace metal grade. (Fisher p/n A509-212 or equivalent)

8.2.3 Hydrochloric acid, HCl, concentrated: Trace metal grade. (Fisher p/n A508SK212 or equivalent)

8.2.4 Potassium permanganate solution, 5% w/v: Prepared by dissolving 50g Potassium Permanganate (Fisher p/n P279-212 or equivalent) in 1000 ml of deionized water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

8.2.5 Potassium persulfate solution, 5% w/v: Prepared by dissolving 50 g Potassium Persulfate (Fisher p/n P282-500 or equivalent)in 1000 ml of DI water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

8.2.6 Sodium chloride-hydroxylamine sulfate solution, 12% w/v: Prepared by dissolving 60 g Sodium Chloride (Fisher p/n) and 60 g of Hydroxylamine Sulfate in 500 ml of DI water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

8.2.7 Stannous chloride (10% $SnCl_2$ w/v in 7% HCl v/v): to a 1000mL volumetric flask dissolve 100 gm Stannous Chloride(VWR part number MK817604) in 70mL conc. HCl. Stir until $SnCl_2$ is completely dissolved. Additional heat may be necessary to get complete dissolution. Once dissolved, dilute to line and cool. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

8.2.8 Aqua regia: In a fume hood, carefully add three volumes of concentrated HCl to one volume of concentrated HNO_3. Prepare fresh daily.

8.3 Stock Standards

8.3.1 Mercury stock standards, 1000 mg/L certified solutions, two sources. One is to be used for the calibration standards and the other for the LCS. (Ultra Scientific ICP-080 and JT Baker 6934-04 or equivalents). Store at room temperature in the metals lab. Expiration dates are given by the manufacturer.

8.4 Calibration Standards:

8.4.1 Intermediate Stock #1 (10,000 ug/L): To a 100 ml volumetric flask add 50 ml DI water and 0.2 ml conc. HNO_3 and 0.2 mL HCl. Transfer 1.0mL of 1000 mg/L Hg stock standard. Dilute to 100 ml with DI water and mix. Prepare fresh daily.

8.4.2 Intermediate Stock #2 (100 ug/L): To a 100 ml volumetric flask add 50 ml DI water and 0.2 ml conc. HNO_3 and 0.2 mL HCl. Transfer 1.0mL of 10,000 ug/L intermediate stock #1. Dilute to 100 ml and mix. Prepare fresh daily.

8.4.3 Use 100mL volumetric flasks. To each, add 50 mL DI water and 0.2mL conc. HNO3 and 0.2 mL conc. HCl. Add the following volumes of 100ug/L intermediate, dilute to volume with DI water, and mix well.

Std Conc. ug/L	Vol. 100ug/L Intermediate #2 Std	Final Volume
0.5	0.5 mL	100 mL
1.0	1.0 mL	100 mL
2.0	2.0 mL	100 mL
4.0	4.0 mL	100 mL
5.0	5.0 mL	100 mL

Using a 25 mL volumetric pipette, transfer 25 mL of each to 50 mL centrifuge tubes. Add 25 mL reagent water to another centrifuge tube for the calibration blank.

8.5 ICV/LCS and CCV: (ICV/LCS from second source, CCV from same source as standards)

8.5.1 Intermediate Stock #1 (10,000 ug/L): To a 100 ml volumetric flask add 50 ml DI water and 0.2 ml conc. HNO_3 and 0.2 mL HCl. Transfer 1.0mL of 1000 mg/L Hg stock standard. Dilute to 100 ml and mix. Prepare fresh daily.

8.5.2 Intermediate Stock #2 (100 ug/L): To a 100 ml volumetric flask add 50 ml DI water and 0.2 ml conc. HNO_3 and 0.2 mL HCl. Transfer 1.0mL of 10,000 ug/L intermediate stock #1. Dilute to 100 ml and mix. Prepare fresh daily.

8.5.3 Check Standard/LCS: To a 100 mL volumetric flask add 50 mL DI and 0.2 ml conc. HNO_3 and 0.2 mL HCl . Add 3.0 mL of the 100 ug/L intermediate stock from the second source standard, dilute to the line and mix.

8.5.4 Using a 25 mL volumetric pipette, transfer 25 mL of each to 50 mL centrifuge tubes.

8.5.4.1 Note: Due to different digestion matrices for aqueous and solid samples, two sets of standards must be prepped to match the matrix for each digestion.

9.0 INSTRUMENT CALIBRATION

In the upper window menu bar select Instrument then calibrate. Enter the calibration information which consists of the standard number and standard concentration in ug/L and press continue. The calibration data will now be available for sample analysis. See section 11.0 for futher calibration instructions.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

	Water	Soil
Preservative:	pH < 2 with HNO_3	4 ^{o}C
Hold Time:	28 days	28 days

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Turn on the Hot Block and allow it to heat to 95^0 C while the samples are being prepared.

11.2 Sample Preparation-Aqueous:

11.2.1 Waters: Using a 25 mL graduated cylinder, transfer 25 mL of sample to a 50 mL polyethylene centrifuge tube. For drinking water analysis, a 25 mL Class A pipette must be used.

11.2.1.1 MS-MSD Prep: Add 0.50 mL of the 100 ug/L intermediate to a 25 mL final volume for a spike concentration of 2.0 ug/L.

11.2.1.2 To each of the samples, MS-MSD, LCS, standards, and blanks add the following (under a hood):1.25 mL conc. H_2SO_4. 0.625 mL conc. HNO_3

11.2.1.3 To all samples, standards, and blanks add 3.75 mL Potassium permanganate ($KMnO_4$) solution.

11.2.1.4 Tightly cap the samples and mix by inverting several times.

11.2.1.5 The purple permanganate color should remain for at least 15 minutes. If it does not, add additional permanganate in 1 mL aliquots until the purple color remains for at least 15 minutes. Record any extra permanganate added in the logbook. The same amount of extra permanganate will have to be added to all other samples and standards.

11.2.1.6 To all samples, standards, and blanks add 2.0 mL Potassium persulfate solution.

11.2.1.7 Place the samples and standards in the Hot Block. Heat at 90-95^0C for 2 hours. Record initial and final Hot Block temperatures in the logbook.

11.2.1.8 Following digestion, remove the samples and place under a hood to cool. Alternately, the racks may be placed in a sink of cold water to hasten the cooling.

11.2.1.9 When the samples are cool, add 1.5 mL Sodium chloride-hydroxylamine sulfate solution to all samples, standards, and blanks. Tightly cap and mix by inverting until samples are clear. Samples are now ready for analysis.

11.3 Sample Preparation-Solids

11.3.1 Weigh triplicate 0.2 gram (approximate) portions from separate areas of the sample container of the untreated sample into a 50 mL polyethylene centrifuge tube with a plastic spatula. Do not use metal spatulas. Record the weight in the mercury prep book. See the subsampling SOP FO-10 for futher instructions on how to obtain a subsample for analysis.

11.3.2 Method Blank and LCS Prep: Weigh 0.50 g of sand blank into each of two 50 mL polyethylene centrifuge tubes. For the LCS, add 0.5 mL of the 100 ug/L second source intermediate stock solution #2.

11.3.3 MS-MSD Prep: Add 0.50 mL of the 100 ug/L intermediate to a 25 mL final volume for a spike concentration of 2.0 ug/L.

11.3.4 To all tubes, add 1.25 mL aqua regia reagent, and heat for 2 minutes in Hot Block at 95 ^{o}C.

11.3.5 Cool, and then add 25 mL of DI water and 3.75 mL of Potassium Permanganate solution to each vial.

11.3.6 Tightly cap all vials and mix by inverting several times.

11.3.7 The purple permanganate color should remain for at least 15 minutes. If it does not, add additional permanganate in 1 mL aliquots until the purple color remains for at least 15 minutes. Record any extra permanganate added in the logbook. The same amount of extra permanganate must be added to all other samples and standards.

11.3.8 Place the samples and standards in the Hot Block. Heat at 90-95 ^{0}C for 30 minutes. Record initial and final Hot Block temperatures in the logbook.

11.3.9 Cool, and then add 1.5 mL of Sodium Chloride-hydroxylamine sulfate to each sample and mix by inverting. The samples should turn clear.

11.4 Instrument Set-up

11.4.1 Power up the M-6000A and autosampler and allow to warm up for one hour.

11.4.2 Turn on lamp and gas supply and allow to warm up for 15 minutes.

11.4.3 Place autosampler tubing into rinse water (1%hydrochloric acid/1%nitric acid solution)

11.4.4 Verify that the sample capillary (inlet insert) is 0.5mm above the gas/liquid separator center post.

11.4.5 Open vents on waste container

11.4.6 Inspect peristaltic pump tubing for wear and flat spots and replace if necessary.

11.4.7 Place the peristaltic pump tubing in their appropriate holes and holder clips. Do not lock shoe clamps at this time.

11.4.8 Initiate M-6000A program by clicking on the M6000 icon, then controls then autosampler page.

11.4.9 Start the autosampler rinse pump by clicking the pump on and the probe down.

11.4.10 Place reagent capillary in a beaker of DI water and start the peristaltic pump in a clockwise rotation.

11.4.11 Lock down the peristaltic shoe clamps.

11.4.12 Inspect liquid flows. The GLS drain should be flowing smoothly with no build up or pulsing of liquid. The waste line from the peristaltic pump to the waste container should be liquid/gas with no vibration. If this is not the case upon inspection, stop

11.4.13 Immediately and change the GLS drain line and/or waste line.

11.4.14 Wet the GLS center post. Pinch the drain line prior to the tee of the peristaltic pump drain tubing. Let two or three liquid bubbles go to the top of the GLS center post and release the drain line. If the liquid does not bubble, then fill the GLS to the top of the center post and release the drain line.

11.4.15 Attach GLS exhaust tube to the GLS

11.4.16 Place reagent capillary in the reagent bottle

11.4.17 Open the appropriate worksheet and verify that the gas flow of the worksheet matches what is listed in the controls, if the flow is not the same make the necessary change and click set gas.

11.4.18 Zero the M-6000A using the autozero. This is under Instrument and then Zero Instrument.

11.4.19 Peak profile the high standard and verify baseline and sample integration times. Do this by clicking on Analysis and then read then standard and then choose the highest standard. If there are any adjustments needed to the peak, refer to the M6000A software manual 5.6.12.

11.5 Analysis

11.5.1 Insert sample labels by clicking on labels and then entering the sample ID numbers.

11.5.2 Right click to enter the QC standards after all the samples are entered. Choose "QC standard" for the CCV and "QC blank for the CCB.

11.5.3 Click on Analysis and then Click on start.

11.5.4 Choose the appropriate box and then click OK.

11.5.5 After analysis, click on file and choose return to main index.

11.5.6 Choose reports

11.5.7 Click on the data tab and choose the data that you want to report

11.5.8 Click on the Reports tab

11.5.9 Click on Write test to file and then enter the LIMS run number and make sure it is saved in the Cetac folder on the I drive.

11.6 Shutdown

11.6.1 Place the reagent capillary in a beaker of 10% nitric acid and cap the reagent bottle. Rinse the system for a minimum of ten minutes.

11.6.2 Place the reagent capillary in a beaker of DI water and rinse the system for one minute

11.6.3 Raise sample probe by clicking on controls then autosampler and click probe up and pump off.

11.6.4 Remove reagent capillary from DI water.

11.6.5 Allow the drain and waste lines to run completely dry

11.6.6 Turn off peristaltic pump

11.6.7 Release peristaltic shoe clamps and release the pump tubing from their holder clips.

11.6.8 Close vents on waste container.

11.6.9 Remove GLS exhaust line from GLS

11.6.10 Turn off gas and lamp

11.6.11 Exit software and run off the autosampler and instrument

12.0 TROUBLESHOOTING

12.1 See Cetac operator's manual for further troubleshooting instructions.

12.2 Preventative maintenance is recorded in the logbook located with the instrument. Follow the recommendations listed in the maintenance section of the Cetac M6000 Operators manual.

13.0 DATA ACQUISITION, CALCULATION AND DATA REDUCTION

Sample Calculations:

Liquid Concentration (ug/L) = A x C

Solid Concentrations (mg/kg) = $\frac{A \times B \times C}{}$

D x E

where A = instrument reading for sample (ug/L)
B = total volume of digestion (L)
C= analyst dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary

$$\textit{Spike Recovery (\%)} = \frac{\text{(Spiked sample concentration – Sample concentration) x 100}}{\text{(Spike amount)}}$$

$$\textit{\%RSD} = \frac{\text{(MS – MSD) x 100}}{\text{(MS + MSD)/2}},$$

where MS = Matrix spike concentration
MSD = Matrix spike duplicate concentration

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer

14.2 LIMS software

14.3 M6000A software

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.

15.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

15.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.

15.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 Non-CLP (Solid Waste and Wastewater-7470A &7471A):

16.1.1 For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 ug/L standards.

16.1.2 ICV (initial calibration verification): Following instrument calibration, analyze the 3.0 ug/L ICV/CCV standard. Control limits are +/-10%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

16.1.3 CCV (Continuing Calibration Verification): Anazlyzed after every 10 samples and at end of run. A 3.0 ug/L standard. Control limits are +/- 20%.

16.1.4 ICB/CCB (initial and continuing calibration blank): After the ICV and any CCV, analyze a blank. The absolute value of the result for the blank must be below the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit or five percent of the measure concentration in the sample. If the result exceeds this, terminate the analysis and correct the problem before proceeding or appropriately qualify the data.

16.1.5 LCS (laboratory control sample): An alternate source standard from the same digestion set as the samples. It is prepared for every 20 samples per medium. Control limits are generated in-house control limits or as specified by client QAPP. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples.

16.1.6 MB (method blank): From the same digestion set as the samples. Blank recovery should be less than the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit or five percent of the measure concentration in the sample. For Army Core of Engineers (ACOE) or Deparment of Defense Quality Systems Manual (QSM) data the criteria is < ½ the reporting limit. If the result is exceeded, reanalyze. If still exceeded, isolate and correct problem, reprep and reanalze the blank and samples associated with the blank, or appropriately qualify results.

16.1.7 MS/MSD (matrix spike/matrix spike duplicate): A MS/MSD is required every analytical run at a frequency of 5% per digestion batch for 7000 series or at a frequency of 10% per digestion batch for 200 series To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. For routine work, use in-house generated limits for the recovery and RPD limits

16.2 CLP-Like Protocol (DOD-QSM/ACOE work):

16.2.1 For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 ug/L standards..

16.2.2 ICV (initial calibration verification): Following instrument calibration, analyze the 3.0 ug/L ICV/CCV standard. Control limits are +/-10%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

16.2.3 ICB (initial calibration blank): Following the ICV, analyze a blank. The absolute value of the result for the ICB must be below ½ the MRL ACOE data, and <2x the MDL for DOD-QSM data. If the result exceeds this, terminate the analysis and correct the problem before proceeding.

16.2.4 LCS (laboratory control sample): Following the calibration verification standard and the calibration verification blank, and every 20 samples thereafter, analyze the 3.0 ug/L alternate source standard. Control limits are specified within the client QAPP or use default limits of 80-120%. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples.

16.2.5 MRL/CRDL standard (Method Reporting limit/contract required detection limit standard): Analyze a standard at the MRL or the CRDLas specified by the client QAPP. Limits are listed within the client QAPP or use a default of 70-130%.

16.2.6 MB (method blank): From the digestion set. If the result exceeds the ½ the MRL, re-digest all samples associated with the MB or appropriately qualify associated results.

16.2.7 CCV (continuing calibration verification): Analyze the 3.0 ug/L calibration standard following every ten samples, and at the end of the analysis. Control limits are +/-20% true value. If the recovery exceeds this, recalibrate and reanalyze all samples back to the last acceptable CCV.

16.2.8 CCB (continuing calibration blank): Analyze a blank following every CCV. The absolute value of the result for the CCB must be below ½ the MRL ACOE data, and <2x the MDL for DOD-QSM data.. If the result exceeds this, reanalyze all samples back to the last acceptable CCB or appropriately qualify results.

16.2.9 Matrix Spike: A matrix spike is required every for every sample delivery group of 20 samples or less. The matrix spike shall be prepared at the time of digestion. Default control limits are +/- 25% true value or or as specified in the projects QAPP, or within limits established by the DOD-QSM . . If recovery is outside of limits, refer to project QAPP or DOD-QSM for further instruction.

16.2.10 Duplicate: A duplicate is required for every sample delivery group of 20 samples or less and is prepared at the time of digestion. For results exceeding five times the MRL, the default control limit is 20% RPD, or as specified in the projects QAPP, or within limits established by the DOD-QSM . For results that are less than five times the MRL, the default control limit is +/- MRL. If precision is outside of limits, refer to project QAPP or DOD-QSM for further instruction.

16.3 SDWA Protocol (245.1)

16.3.1 For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 ug/L standards

16.3.2 ICV (initial calibration verification): Following instrument calibration, analyze the 3.0ug/L ICV/CCV standard. Control limits are +/-5%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

16.3.3 ICB (initial calibration blank): Following the ICV, analyze a blank. The absolute value of the result for the ICB must be below the MDL for the method. If the result exceeds this, terminate the analysis and correct the problem before proceeding or appropriately qualify the data.

16.3.4 LCS (laboratory control sample): Following the calibration verification standards, and every 20 samples thereafter, analyze the 3.0 ug/L alternate source standard. Control limits are generated control limits. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples associated with the LCS.

16.3.5 MB (method blank): From the same digestion set as the samples. Limits: the highest of the MDL, 5% of the measured concentration in the sample, or 5% of the regulatory limit for that analyte. If the results exceed this, terminate the run, reprep and reanalyze all samples associated with MB or appropriately qualify the data.

16.3.6 CCV (continuing calibration verification): Analyze the 3.0ug/L calibration standard following every ten samples, and at the end of the analysis. Control limits are +/-10% true value. If the recovery exceeds this, recalibrate and reanalyze all samples back to the last acceptable CCV.

16.3.7 CCB (continuing calibration blank): Analyze a blank following every CCV. The absolute value of the result for the CCB must be below the highest of the MDL, 5% of the measured concentration in the sample, or 5% of the regulatory limit for that analyte. If the result exceeds this, reanalyze all samples back to the last acceptable CCB or appropriately qualify results.

16.3.8 MS/MSD (matrix spike/matrix spike-duplicate): A MS/MSD is required every analytical run at a frequency of 10% per matrix type. For digested samples, appropriate samples are designated and spiked at the time of the digestion. Control limits are in-house limits.

16.4 Data Review Prior to Shutdown: Check results for the following criteria. It may be possible to correct some problems within the same analytical run.

16.4.1 ICV/CCV, LCS, MB, MRL acceptance. See above for corrective action.

16.4.2 Results over the calibration range of 5 ug/L. Dilute affected samples within calibration range with dilution blank.

16.4.3 MS-MSD recovery outside of acceptance range. If both are out of range, but all other CCV, CCB, and LCS associated with the MS/MSD are within specs, then the problem is matrix related-not instrument related. Sample data may be reported but flagged with the appropriate comment.

16.5 Analysis data review checklist- the checklist will be completed by the analyst and the data reviewer and attached to the analytical data package.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, EPA, SW-846, Methods 7470A, 7471A.
17.2 Statement of Work for Inorganic Analysis, USEPA Contract Laboratory Program,. ILM04.0.
17.3 Methods for the Determination of Metals in Environmental Samples. EPA/600/R94/111, Method 245.1

17.4 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, January, 2006.

17.5 Cetac-M6000A operations manual and software manual.

18.0 TABLES

18.1.1 Mercury Spike Prep

COLD VAPOR Element	Spike Amt. mL	Spike Solution Supplier	Stock Conc. ug/L	Final Vol. mL	Expected Conc. ug/L
Hg	0.50	Ultra 1000 mg/L	100.0	25.0	2.0

Standard Quality Control Requirements and Corrective Action

QC Type	Frequency	Conc. Level	*Acceptance Criteria*	*Corrective Action*
ICal	Each time the instrument is set up. The ICal consists of five standards and a blank.	0 + 0.5 – 5.0 ug/L	Correlation coefficient of .995 or greater	Terminate analysis, correct problem and recalibrate.
ICV	Immediately after the ICal	3 ug/L	Second source standard, SDWA: 95-105% SW846:90-110% ACOE- see client QAPP	Reanalyze once, if still unacceptable terminate analysis, correct problem and recalibrate
ICB	Immediately after the ICV	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. ACOE: ½ the MRL DOD-QSM: < 2x the MDL	Reanalyze once, if still unacceptable terminate analysis, correct problem and recalibrate.
LCS	1 per batch of ≤ 20 samples per matrix per day	mid cal. Range	SDWA; 90-110% SW846; 80-120% ACOE- see client QAPP	Reanalyze once, if still unacceptable terminate analysis, correct problem and reanalyze all associated samples. High bias is acceptable for associated samples that have results less than the MDL for routine work or the MRL for ACOE work.
CCV	After every 10th sample and at the end of the analytical sequence	mid cal range	SDWA; 90-110% SW846; 80-120% ACOE- see client QAPP	Reanalyze once, if still unacceptable recalibrate and reanalyze all samples back to the last acceptable CCV or ICV. High bias is acceptable for associated samples that have results less than the MDL for routine work or the MRL for ACOE work.
CCB	Immediately following each CCV	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. ACOE: ½ the MRL DOD-QSM: < 2x the MDL	Reanalyze once, if still unacceptable reanalyze all samples back to the last acceptable CCB or appropriately qualify results.

MS-MSD (routine work) or MS-DUP (ACOE)	5% of samples per matrix per day	See attached spike chart	≤ ± 20%, Applicable when spike level is >25% of original analyte level in the sample and RPD ≤ ± 20% or as specified in DOD-QSM or ACOE QAPP	Perform PDS
Post Digestion Spike (PDS)	Upon failure of MS or per batch for ACOE work	Same level as MS	85-115%	Qualify data as matrix interference or perform MSA
Method Blank	1 per batch of 20 samples	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. DOD-QSM/ACOE: <1/2 the MRL	Investigate and isolate possible source and correct problem; then reanalyze all associated samples, if possible, or qualify data (B)

GFAA / FLAA / CVAA Data Review checklist			Method: 200.9 7000 series AA 245.1 7470/7471		
Instrumentation: PE SIMAA 6000	VARIAN SPECTRAA10		CETAC HG ANALYZER		
Analysis Date:	Data File:		Date Review:		**Analyte:**_____
Cal Std ID:	LIMS # :	Analyst:	Reviewer:		Approved? Yes No
Calibration Parameters -	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV 90-110% 95-105%					
3) ICB ABS of LOD or RL					
6) CRA - (2X CRDL or LOD) 80-120%					
7) CCV1/CCB1- 90-110% / 80-120%					
8) CCV2/CCB2 ABS of LOD or RL					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
12) CCV6/CCB6					
Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	**Comments:**
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					

Environmental Chemistry Branch
Engineer Research and Development Center

TITLE:

DETERMINATION OF NITROCELLULOSE IN SOIL SAMPLES BY BASIC HYDROLYSIS AND ION CHROMATOGRAPHY OF NITRATE AND NITRITE.

SOP NUMBER:

PREPARED BY: _____

REVIEWED BY: _____
Technical Specialist Date

_____
QA Officer Date

_____
Branch Chief Date

SOP MANUAL CONTROL NO.: _____

1.0 Scope and Application

1.1 This method is intended for the extraction and measurement of nitrocellulose in soil samples. This method is limited to use by analysts experienced in handling and analyzing explosive residues.

1.2 This method can be used to measure nitrocellulose in soils and sands that can be air dried and ground in a mortar and pestle.

1.3 Nitrocellulose of greater than 20 mg/kg can be measured by this method.

2.0 Method Summary

2.1 A dried and ground sample is extracted with water and ethanol to remove inorganic nitrate and nitrite and ethanol soluble explosives.

2.2 The remaining solids are extracted with acetone.

2.3 Basic hydrolysis is used to liberate nitrate and nitrite from the nitrocellulose. The solution is neutralized.

2.4 The nitrate and nitrite are measured by ion chromatography. Theoretical nitrocellulose is found by calculation.

3.0 Health and Safety

3.1 The acetone used as the extraction solvent is flammable. It should not be used near sparks or an open flame.

3.2 The hydrolysis is performed at high temperature in sealed plastic tubes. No acetone should remain in the tube before hydrolysis. The hydrolysis must be carried out in a hood behind a shield.

4.0 Sample Preservation, Containers, Handling, and Storage

4.1 Grab samples must be collected and stored in glass containers. Follow conventional sampling procedures.

4.2 Samples must be kept below 4°C and in the dark from the time of collection through analysis except when being dried.

4.3 Soil samples should be air dried to constant weight at room temperature. The nitrocellulose content of dried and ground soil samples is stable.

5.0 Interferences and Potential Problems

5.1 All acetone should be evaporated before hydrolysis to avoid excessive pressure in the hydrolysis tube.

5.2 Nitrocellulose is not stable in acetone. Minimize the time that the extracts are in acetone.

6.0 Equipment/Apparatus

6.1 Balance - ± 0.1 mg.

6.2 Centrifuge - International Centrifuge Model 2K or equivalent

6.3 Oven - Forced air, without heating.

6.4 Tube Rotator - Scientific Equipment Products CO Model 60448 or equivalent

6.5 Hot Plate

6.6 Materials

6.6.1 Finnpipettes – 1 mL, 5 mL.

6.6.2 9-cm evaporating dish.

6.6.3 Custom-made air and CO_2 manifold.

6.6.4 Mortar and pestle - Steel.

6.6.5 Sieve - 30 mesh.

6.6.6 Disposable centrifuge tube - 50 mL with a screw cap.

6.6.7 Filter Paper – Whatman #40 11 cm or equivalent.

6.6.8 pH Paper (pH 7-14)

6.6.9 Acetone-rinsed and dried sand for LCS and blanks

6.7 Ion analyzer - Dionex DX-100 or equivalent.

7.0 Reagents

7.1 HPLC grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without affecting the accuracy of the determination.

7.2 General

7.2.1 Nitrocellulose – Aldrich, CAT# 435031-250G (St. Louis)

7.2.2 Reagent water - Use water from the Barnstead Nanopure system in the inorganic prep area.

7.2.3 Acetone – reagent grade

7.2.4 Ethanol – reagent grade

7.2.5 Sodium Bicarbonate - reagent grade.

7.2.6 Sodium Carbonate - reagent grade.

7.2.7 Concentrated Sulfuric Acid - reagent grade.

7.3 Stock Standard Solutions

7.3.1 Dry the nitrocellulose standard to constant weight in a vacuum desiccator in the dark. Place 416.6 mg (weighed to the nearest 0.1 mg) into a 100 mL volumetric flask and dilute to volume with acetone. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 4166 mg/L). Use the stock solution within one week.

7.3.2 Nitrite Standard - Weigh 0.1500 g Sodium Nitrite and dilute to 100 mL in water.

7.3.3 Nitrate Standard - Weigh 0.1630 g Potassium Nitrate and dilute to 100 mL in water.

7.4 Eluent: dilute 0.7632 g Na_2CO_2 and 0.5712 g $NaHCO_2$ to 4.00 L in a 4 L solvent bottle.

7.5 Regenerant: add 3.0 mL of concentrated sulfuric acid to 4.00 L water in a 4 L solvent bottle.

7.6 Working Standards:

7.6.1 Nitrite/Nitrate Intermediate Standard - Combine 1.00 mL Sodium Nitrite stock solution with 1.00 mL Sodium Nitrate stock solution and dilute to 100 mL in water (10µg/mL).

7.6.2 Pipet 5.00, 2.00, 1.00, and 0.50 mL of Nitrite/Nitrate Intermediate Standard into individual 10 mL volumetric flasks and dilute to volume with water.

8.0 Procedure

8.1 Place about 200 grams of wet sample into a 9-cm evaporating dish. Dry overnight in a forced air oven with the heating turned off.

8.2 Grind the sample in a steel mortar and pass it through a 30 mesh screen.

8.3 Weigh an appropriate amount of sample into a 50-mL centrifuge tube based on projected contamination levels (typically 10 g unless high level contamination is present). Label the sample identities on the tubes.

8.3.1 Weigh out 10 g of rinsed sand for LCS and blanks. Weigh out duplicate samples and duplicate spiked samples for each project in the run. Weigh out additional duplicate and spike sets for each set of more than 20 samples.

8.3.2 Add 1.00 mL of intermediate standard to the spike samples. This will result in a final total concentration of 20 ppm nitrate/nitrite.

8.3.3 Dry spiked samples under a gentle flow of forced air until all traces of acetone are gone (about 30 min). Pipet 15 mL of reagent water into the tubes.

8.4 Rotate the tubes on the clinical rotator for 15 minutes.

8.5 Centrifuge the tubes at maximum rpm in the International Centrifuge for 15 minutes.

8.6 Decant the supernatant water and dispose of it.

8.7 Pipet 15 mL of ethanol into the tubes. Cap and shake the tubes to break up the pill of sample in the tube's bottom.

8.8 Rotate the tubes on the clinical rotator for 15 minutes.

8.9 Centrifuge the tubes at maximum rpm in the International Centrifuge for 15 minutes

8.10 Decant the supernatant ethanol and dispose of it.

8.11 Pipet 15 mL of acetone into the tubes. Cap and shake the tubes to break up the pill of sample in the tube's bottom.

8.12 Rotate the tubes on the clinical rotator for 15 minutes.

8.13 Centrifuge the tubes at maximum rpm in the International Centrifuge for 15 minutes.

8.14 Decant the supernatant acetone into a new, labeled 50 mL screw top centrifuge tube.

8.15 Repeat steps 8.11 through 8.14, adding the second acetone rinse to the first, for a final acetone volume of 30 mL in the centrifuge tubes.

8.16 Evaporate the acetone to dryness under a gentle flow of forced air, usually overnight.

8.17 Add 5 mL 5N aqueous NaOH to dried extracts. Cap the tubes tightly and place them in a boiling water bath for 8 minutes.

8.18 Cool the tubes for at least 30 minutes. Dilute to final volume of 20.0 mL with reagent water.

8.19 Gently bubble compressed carbon dioxide through the extracts until pH is less than 8 (approximately 25 minutes).

8.20 Dilute once more with reagent water to a final volume of 25 mL.

8.21 Inject 50 µl of the sample on the Dionex channel A ion analyzer. Retention times for nitrite and nitrate in the sample extracts may shift based on bicarbonate concentration.

9.0 Calculations

9.1 RESPONSE FACTORS: Response factors are calculated for nitrite and nitrate at five levels. The response factor, RF, equals the peak height, R, divided by the concentration, C, in µg/mL.

$$RF = R/C$$

9.2 ANALYTE CONCENTRATION: Nitrite and Nitrate concentration, C, in the extract are found by dividing the response for the sample by the response factor.

$$C = R/RF$$

9.3 The concentration of nitrocellulose [NC] is found by first calculating the total ppm of nitrite and nitrate, as given by the instrument.

9.4 Since analytical grade nitrocellulose is about 12.0% nitrate by mass, divide the total ppm of nitrite and nitrate by 0.12.

9.5 To correct for sample dilution into liquid, divide by 40. (1000/25 mL final sample volume = 40)

9.6 The concentration of nitrocellulose [NC] in the original sample in mg/kg is then calculated by correcting for sample mass to kg (for a 10 g sample size, multiply by 100).

10.0 Quality Assurance/Quality Control

10.1 A preparation batch of samples is defined as a group of up to twenty field samples of similar matrix type that are extracted at the same time using the same reagents for the same analysis. In addition to the twenty samples, each batch also contains, at a minimum, a method blank, a matrix duplicate (MD), a laboratory control sample (LCS), and a matrix spike and matrix spike duplicate (MS/MSD).

10.1.1 The laboratory must, on an ongoing basis, analyze a method

blank, a matrix spike, and a matrix spike duplicate/duplicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

10.1.2 A minimum of one duplicate sample shall be run with each analytical batch. If the samples are generally non-detect samples, a matrix spike duplicate must be run with the analytical batch.

10.2 A method blank must be prepared and analyzed with each batch of samples. The method blank shall be prepared from the acetone-rinsed dry sand (usually 10 g).

10.3 The LCS sample must be prepared and analyzed with each batch of samples. The LCS shall be spiked with the appropriate LCS spiking solution. Weigh 10 g of acetone-rinsed dry sand and spike with 1 mL of the nitrocellulose standard. Control charts will be maintained for the LCS. The order of preference for spiking levels is as follows:

- If an action level exists, spike at this level
- If the above condition does not apply, spike at a level between the low and the mid-point of the calibration curve.

10.4 The use of the matrix spike and matrix spike duplicate will depend in part on the role of the laboratory. When the laboratory serves as the primary laboratory, then site-specific documents should be consulted. The sample to be used for the MS/MSD may be specified in the field. This previously designated sample would then be spiked with the site-specific target analytes at a concentration equivalent to the site action level. If this information was not specified or unknown, then the laboratory would choose a representative sample from each batch of samples analyzed. If samples from multiple sites were to be analyzed in the same batch, then multiple sets of MS/MSDs may be required.

When the laboratory serves as the QA laboratory, the above scenario may not be practical to implement. If the site-specific requirements are unknown and samples from multiple sites are analyzed in the same batch, then the laboratory should select a single sample for spiking. The order of preference for spiking levels is as follows:

- If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two
- If an action level exists, spike at this level
- If neither of the first two conditions apply, spike at a level that corresponds between the low and the mid-point of the calibration curve.

10.5 The use of the matrix duplicate may also depend in part on the role of the laboratory. The selection of a matrix duplicate will be performed as

described for the MS/MSD in Section 10.4.

10.6 The extraction analyst must demonstrate proficiency in performing the analysis as outlined in SOP No. Q-016-ECB-QC, Technical Training. Method proficiency must be redemonstrated whenever a major method modification is made.

11.0 Data Validation

11.1 The extraction analyst is responsible for verifying the information recorded on the extraction log is complete and accurate.

11.2 Additional levels of review are performed as described in SOP No. Q-024-ECB-DR, Data Reduction and Review/Validation (In-House/Contractor Data).

12.0 Waste Disposal

12.1 Solvent wastes are generated during this procedure. All waste disposal procedures must comply with all federal and local regulations. Refer to the Environmental Chemistry Branch Waste Management Plan.

12.2 Extracted soil samples will be drummed in their centrifuge tubes and processed by a hazardous waste disposer.

12.3 Decanted ethanol will be drummed and processed by a hazardous waste disposer.

12.4 Other wastes with be drained into the sanitary sewer system.

13.0 References

13.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition (1995), Method 2540C.

13.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Method 3510B, Revision 2, September 1994.

13.3 EPA-600/4-82-029 Methods for Chemical Analysis of Water and Wastes.

13.4 SOP No. Q-005-ECB-SH, Sample Receipt, Login and Storage.

13.5 SOP No. Q-012-ECB-GL, Glassware Cleaning.

13.6 SOP No. Q-016-ECB-QC, Technical Training and Personnel Qualifications.

13.7 SOP No. Q-024-ECB-DR, Data Reduction and Review/Validation (In-House/Contractor Data).

13.8 Environmental Chemistry Branch Waste Management Plan.

13.9 SOP No. Q-008-ECB-GL, Reagent Water Generation and Quality Monitoring.

13.10 SOP No. Q-004-ECB-QC, Nonconformances and Corrective Action.

13.11 SOP No. Q-023-ECB-QC, Method Equivalency Demonstration.

13.12 "Analytical Method For The Determination Of Nitrocellulose In Water" T.F. Jenkins (1989) Private communication.

13.13 Hubaux, A. and G. Vos (1970) Decision and detection limit for linear calibration curves. Analytical Chemistry, 42:849-855.

13.14 Barkley, J.J. and D.H. Rosenblatt (1978) Automated nitrocellulose analysis. U.S. Army Medical Bioengineering Research and Development Laboratory, Technical Report 7807, ADA067081.

13.15 Helton, D.O. (1976) Chemical and physical characterization of nitrocellulose fines. U.S. Army Medical Research and Development Command, ADA036151.

THE SEQUENCE OF BLANKS, BLANK SPIKES DUPLICATES AND SPIKED DUPLICATE SAMPLES

Ion Chromatography runs will be sequenced as follows;

1. Instrument blank (IBL)
2. Std. 0.5 µg/mL
3. Std. 1.0 µg/mL
4. Std. 2.0 µg/mL
5. Std. 5.0 µg/mL
6. Std. 10.0 µg/mL
7. Method blank
8. Spiked method blank
9. Control sample
10. Matrix analysis (MA) for background (sample i)
11. Matrix analysis duplicate (MAD)
12. Spiked matrix analysis (MS)
13. Spiked matrix analysis duplicate (MSD)
14. Sample (ii)
15. Sample (iii)
16. Sample (iv)
17. Sample (v)
18. Sample (vi)
19. Sample (vii)
19. Sample (viii)
20. Sample (ix)
21. Sample (x)
22. Std 2.0 µg/mL
23. IBL

REPORTING REQUIREMENTS AND DATA VALIDATION. Reports will include a minimum of a blank, blank spike, sample duplicate, matrix spike, and matrix spike duplicate. RPD must be below 25%. Percent recovery must be between 50 and 100%.

CT Laboratories
Baraboo Laboratory Division

Title: **Semi-Volatile PCB as Aroclor Analysis**

SOP Number: **8082**

Written by: _____

Technical Review by: _____

Approved by: _____
Quality Assurance Manager

Approved by: _____
Laboratory Director

SOP Manual Control Number: _____

1.0 SCOPE/APPLICATION

1.1 Method SW-846 8082 is used to determine the quantitation of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid and aqueous matrices. Capillary columns are employed with electron capture detectors (ECD). Target Aroclors determined by this method are listed in Table 1.0 with current method detection limits.

1.2 This method is a dual column analysis. Each column uses the same QC criteria for the reporting of analytical results. The analytical columns are joined to a single injection port. The secondary chromatographic column is used to confirm or to report final results in conjunction with the primary column.

1.3 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. Multiple Aroclors can be present within a sample. The analyst will report all Aroclors present with in the sample. The analyst will address the multiple Aroclors in the case narrative.

1.4 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

Table 1.0
Aroclors with Method Detection Limits and Limits of Quantitation

Aroclors	***Water MDLs (ug/L)***			***Soil MDLs (mg/kg)***	
	MDLs	***LOQs***		***MDLs***	***LOQs***
1016	0.114	0.379		0.009	0.031
1221	0.071	0.236		0.012	0.041
1232	0.140	0.468		0.014	0.047
1242	0.096	0.322		0.010	0.034
1248	0.118	0.393		0.009	0.029
1254	0.076	0.253		0.003	0.010
1260	0.115	0.384		0.006	0.019
1262	0.140	0.468		0.006	0.021
1268	0.035	0.116		0.005	0.018

Table 1.1
Aroclors with Method Detection Limits and Limits of Quantitation
For Low Level Soils

Aroclors	***Soil MDLs (mg/kg)***	
	MDLs	***LOQs***
1016	0.003	0.012
1221	0.005	0.017
1232	0.006	0.019
1242	0.006	0.021
1248	0.004	0.012
1254	0.002	0.008
1260	0.003	0.009
1262	0.002	0.007
1268	0.003	0.010

Table 1.2
Aroclors and Retention Times

Aroclor Name	**RT Peak#1**	**RT Peak#2**	**RT Peak#3**	**RT Peak#4**	**RT Peak#5**
1016	0.90	1.21	1.44	1.49	1.85*
1221	0.83	0.87*	0.90	---	---
1232	0.90	1.21	1.44	1.49	1.76*
1242	0.90	1.21	1.44	1.49	1.64*
1248	1.21	1.37	1.44	1.49	2.24*
1254	2.10	2.29	2.53*	2.85	3.32
1260	2.84	3.32	3.49	4.22*	4.69
Additional Aroclors	**RT Peak#1**	**RT Peak#2**	**RT Peak#3**	**RT Peak#4**	**RT Peak#5**
1262	3.06	3.32	3.49	3.71	4.93*
1268	3.49	3.80	4.28	4.32	4.53*

Note: Retention time shifts can occur when instrument maintenance is performed. Shifts in the retention times are reflected in the analytical method. "*" Indicates unique calibration peak for that aroclor.

Table 1.3
Laboratory Control Sample Limits

	Inhouse					**QSM**			
	Water (ug/L)		**Soil (mg/kg)**			**Water (ug/L)**		**Soil (ug/kg)**	
Analyt	**Lower Limit**	**Upper Limit**	**Lower Limit**	**Upper Limit**		**Lower Limit**	**Upper Limit**	**Lower Limit**	**Upper Limit**
1016	58	128	64	123		25	145	40	140
1260	62	126	60	125		30	145	60	130
TCMX	42	148	66	124		---	---	---	---
DCB	55	155	62	149		40	135	60	125

Table 1.4
Matrix Spike/Matrix Spike Duplicate Control Limits

	Inhouse						QSM					
	Water (ug/L)			Soil (mg/kg)			Water (ug/L)		RPD	Soil (ug/kg)		RPD
Analyt	Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit		Lower Limit	Upper Limit	
1016	57	133	26	46	150	23	25	145	30	40	140	30
1260	53	139	27	48	143	21	30	145	30	60	130	30
TCMX	32	159	---	39	148	---	---	---	---	---	---	---
DCB	50	149	---	35	159	---	40	135	---	60	125	---

Note: Information on recovery limits and RPDs are generated though StarLIMS. The information tables are stored in H:\QC\charting.

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous and soil matrices (reference methods SW846-3535 and 3545), concentration techniques that are suitable for preparing the extract, and the quantitative/qualitative analysis for the determination of Aroclors by method SW846-8082.

2.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method applies for aqueous samples extracted solid phase extraction (SPE) SW846-3535. Method applies for soil/sediment, and solid waste samples extracted by standard solvent extraction methods utilizing pressurized extraction techniques as heated pressurized fluid extraction SW846-3545. This method includes the extraction for waste dilution samples.

2.3 The resultant extract is chemically dried and concentrated using a TurboVap system and/or Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

2.4 Extracts for Aroclor analysis can be subjected to a variety of cleanup steps, depending on the nature of the matrix interferences and target analytes. Suggested cleanup steps include Florisil (Method 3620), sulfur cleanup (Method 3660), sulfuric acid cleanup (Method 3665), and Gel-Permeation Chromatography (GPC) cleanup (Method 3640A). See attachments; I, II, III, and VI. After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with dual capillary columns and ECD detectors.

2.5 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

3.0 DEFINITIONS

3.1 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.2 Laboratory Control Spike (LCS): Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with 1016/1260 Aroclors (unless a specified Aroclor(s) is requested) and carried through the complete sample preparation and analytical procedure. The control spike is used to document the ability of an analyst to generate acceptable precision and bias to verify the analytical system performance, and to document method accuracy for each matrix.

3.3 Laboratory Control Spike Duplicate (LCSD): A duplicate Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with 1016/1260 Aroclors (unless a specified Aroclor(s) is requested) and carried through the complete sample preparation and analytical procedure. The control spike duplicate is used to document the precision and bias of a method. This default QC type is only performed when insufficient amount of sample is available for performing a matrix spike and matrix spike duplicate.

3.4 Matrix Spike (MS): An aliquot of sample spiked with 1016/1260 Aroclors (unless a specified Aroclor(s) is requested). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): Intra-laboratory split samples spiked with 1016/1260 Aroclors (unless a specified Aroclor(s) is requested). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.6 Method Reporting Limit (MRL): The method reporting limit is a threshold value below which the laboratory reports a result as non-detect. The highest value reported for the method reporting limit is dependant upon project-specific action or decision levels. Method reporting limits are adjusted based on the sample matrix and any sample dilution/concentrations when necessary.

3.7 Method Detection Limit (MDL): The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. It is determined from analysis of a sample in a given matrix containing the analyte. Depending on project specifics, MDLs will be in

the form of a MDL check standard or the MDL is subject to extraction procedures. An acceptable MDL standard check must produce a signal at least three times the instrument's noise level. Also, a MDL check injection must be made after each major instrument repair or changing of instruments (prior to sample analysis) to determine the performance sensitivity of the analysis.

3.8 Surrogate (SURR): An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. It's use is to monitor the performance of the extraction, cleanup (as needed), the analytical system, and the effectiveness of the method. The surrogates are; 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl.

3.9 Initial Calibration (ICAL): An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of Aroclors 1016/1260. This relationship is depicted graphically, and referred to as a "calibration curve". Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte. Alternative ICALs for Aroclors can be substituted for the 1016/1260 ICAL if specified by project or program.

3.10 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed.

3.11 Continuing Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis.

3.12 Solid Phase Extraction (SPE): The separation ability of solid phase extraction is based on the preferential affinity of desired or undesired solutes in a liquid, mobile phase for a solid, stationary phase through which the sample is passed. Impurities in the sample are either washed away while the analyte of interest is retained on the stationary phase, or vice-versa. Analytes that are retained on the stationary phase can then be eluted from the solid phase extraction cartridge with the appropriate solvent.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis

by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems will be necessary. Refer to each method for specific guidance on quality control procedures.

4.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, will cause degradation of certain analytes.

4.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample will be necessary.

4.5 Elemental sulfur (S_8) is readily extracted from soil samples. High concentrations of sulfur will cause chromatographic interferences in the determination of Aroclors. Sulfur can be removed through cleanup procedures such as Method 3660.

5.0 SAFETY

5.1 Protective clothing; safety glasses, gloves, apron and/or lab coat, long pants, and protective shoes, will be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure will utilize appropriate laboratory safety systems.

5.2 The toxicity of chemicals used in this method has not been precisely defined. Each chemical will be treated as a potential health hazard, and exposure to these chemicals will be minimized.

6.0 APPARATUS & MATERIALS

Apparatus:

6.1 Gas Chromatograph- An analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including syringes, analytical columns, electron capture detectors, auto sampler, electronic pressure control, and EZ Chrom Elite data acquiring system.

6.1.1 Carrier Gas: Hydrogen at 27.6 initial psi at 12.0 ml/min.
Mode: Ramped Flow
Initial flow: 12 ml/min. (hold for 1.0 min)

Ramp: 2.5 ml/min.
Final: 15.1 ml/min. (hold for 0 min.)
Ramp: 3.2 ml/min.
Final: 24 ml/min. (hold for 2.0 min.)

Injection Volume: 1 ul
Injector: 250 ^{o}C
Pulse Pressure: 37 psi
Mode: Pulse Split
Pulse Time: 0.2 min.
Split Ratio: 1.0
Split Flow: 35.9 ml/min.
Total Flow: 79 ml/min

Detector (s): 300 ^{o}C
Mode: Constant Flow
Make-up Gas: Nitrogen at 40 mL/min.

Oven: Initial – 180 ^{o}C (hold for 0.4 minutes)
Ramp – 10.1 ^{o}C/min.
Final - 192 ^{o}C (hold for 0 minutes)
Ramp – 13 ^{o}C/minute
Final - 220 ^{o}C (hold for 0 minutes)
Ramp – 30 ^{o}C/minute
Final - 291 ^{o}C (hold for 0.1 minutes)

Note: Instrument operating parameters are subject to change to improve overall chromatography. (Changes are noted in the Instrument Maintenance Log Book).

6.2 Analytical Column Pair:

6.2.1 30m x 0.32 mm ID bonded with 5% phenyl polysiloxane / 95% dimethylsiloxane, 0.25 um. (ZB-5, part # 7HM-G002-11 or equivalent).

6.2.2 30m x 0.32 mm ID bonded with 14% Cyanopropylphenyl/86% dimethylplysiloxane, 0.25 um (ZB-1701, part # 7HM-G006-11 or equivalent).

6.3 Solid Phase Extraction Unit (SPEU). 3 station manifold assembly (47mm). (UCT: ECUCTVAC347).

6.4 House Vacuum.

6.5 Dionex ASE 200.

6.6 TurboVap; autoconcentrator

6.7 Organomation Nitrogen blow down concentrator.

6.8 Analytical balance capable of accurately weighing to the nearest 0.01 gram (for sample preparation).

6.9 Oven.

Glassware:

6.10 Solid Phase Extraction Apparatus (SPEA)
- 6.10.1 1L gradutated funnel (Fisher # 09-753-26B).
- 6.10.2 Stainless Steel Support Screen (Fisher # 09-753-29A).
- 6.10.3 Clamp (Fisher # 09-753-31A).
- 6.10.4 Glass Support Base (UCT: ECQSB47).

6.11 Kuderna-Danish (K-D) apparatus:
- 6.11.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).
- 6.11.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).
- 6.11.3 Synder column- Three-ball macro (Fisher # K503000-0121).
- 6.11.4 Springs or Teflon clamps to attach concentrator tube to evaporation.

6.12 Graduated cylinder (Class A) - 1000 mL. (Fisher 08-559G).

6.13 Beaker - 250 mL and 600 mL.

6.14 Vials - 2.0 Ml, 12ml, and 60 mL screw cap vials with Teflon lined caps (Fisher # 03-377-10B).

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6.15 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

6.16 Funnels – glass.

6.17 Volumetric flask – (Class A) 10, 25, 50, and 100 mL.

6.18 Syringes 10 uL, 100 uL, 500 uL, and 1,000 uL.

6.19 TurboVap concentrator tubes. (Caliper # 45817).

6.20 40ml VOA vials (QEC # 2112-40ml).

6.21 1L amber glass jar (QEC # 6122-0032).

Reagents:

6.22 Deionized water (Milli-Q processed).

6.23 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212). Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil.

6.24 Silica sand- hydrocarbon free. Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil.

6.25 Methylene chloride, pesticide grade.

6.26 Acetone, pesticide grade.

6.27 Methanol, pesticide grade.

6.28 Hexane, pesticide grade.

6.29 Isopropanol, pesticide grade.

6.30 Hydrogen (99.995% purity or greater)

6.31 Nitrogen (99.995% purity or greater).

6.32 Sulfuric Acid (Certified ACS)/Deionized Water-1:1(v/v)..

6.33 Sodium Hydroxide- 10 N (Certified ACS).

6.34 Diatomaceous earth (JT Baker # 1939-01).

6.35 House Air

Materials:

6.36 Boiling chips, carborundum, approximately 10/40 mesh (methylene chloride rinsed) (G. Frederick Smith Chemical Company, Catalog #198).

6.37 pH indicator paper- pH 0-14 (Whatman Catalog #2613991).

6.38 Empore Extraction Disk (SPE): (Phenomenex # AHO-2540).

6.39 Dionex ASE Filters (Catalog #049458).

6.40 Filter- Glass Microfiber 12.5 cm GF/A (Whatman # 1827-125).

6.41 Aluminum foil.

6.42 Spatulas- stainless steel.

7.0 STANDARDS AND SPIKES

7.1 Preparation of standards is documented in the Pest/PCB standards logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards, once opened, expire within six months or sooner if routine QC indicates a problem and, are not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire within six months and are not to exceed the opened date of the stock standard and or exceed manufactures expiration date.

7.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Stock standards, once opened, expire within six months and are not to exceed the manufactures expiration date. The following list of stock standards are commercially prepared certified standards, such as;

Aroclor 1016/1260: Restek Part # 32039 at 1000 ug/mL.
Aroclor 1016: Restek Part # 32006 at 1000 ug/ml.
Aroclor 1221: Restek Part # 32007 at 1000 ug/ml.
Aroclor 1232: Restek Part # 32008 at 1000 ug/ml.
Aroclor 1242: Restek Part # 32009 at 1000 ug/ml.
Aroclor 1248: Restek Part # 32010 at 1000 ug/ml.
Aroclor 1254: Restek Part # 32011 at 1000 ug/ml.
Aroclor 1260: Restek Part # 32012 at 1000 ug/ml.
Aroclor 1262: Restek Part # 32409 at 1000 ug/ml.
Aroclor 1268: Restek Part # 32410 at 1000 ug/ml.
Surrogate Mix: Restek Part # 32000 at 200 ug/ml.

7.3 Intermediate Working Stock Standards: The Aroclor intermediate standards are prepared at an optimum level from the preparation of the working stock standard. Each Aroclor with surrogate are prepared at an optimum level for the intermediate stock standard. The concentration of each Aroclor is 10 ug/ml with surrogate concentrations of 0.5 ug/ml in hexane. The following is a list of each Aroclor.

Aroclor 1016	Aroclor 1254
Aroclor 1221	Aroclor 1260
Aroclor 1232	Aroclor 1262
Aroclor 1242	Aroclor 1268

Table 2.0
Intermediate Working Stock Standard Concentration

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	1000	0.100	10.0	10.0
Surrogate	200	0.025	10.0	0.5

7.4 Calibration standards: PCB are to be determined as Aroclors, external calibration techniques will be used. An initial calibration is performed using a minimum of a five point calibration curve for Aroclors 1016/1260. These standard mixes will include many of the peaks represented in the other Aroclor mixtures. Standards for the other Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor (CF) for that specified Aroclor. The single-point calibration standard level is marked with an asterisk. In situations where only a few Aroclors are of interest for a specific project or program, the analyst will employ a minimum of a five point initial calibration of each of the Aroclors of interest. Alternative standard concentrations for the calibration curve can be prepared to meet client's or program's specified criteria.

Level 1	0.030 ug/ml
Level 2	0.050 ug/ml
Level 3	0.200 ug/ml
Level 4*	0.500 ug/ml
Level 5	1.000 ug/ml
Level 6	1.200 ug/ml

Table 2.1
Calibration Points for the Aroclor(s) Linearity

Linearity Points	Spike Concentration (ug/ml) Aroclor(s)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml) Aroclor(s)	Final Concentration (ug/ml) Surrogate**
1	10.0	0.030	10.0	0.03	0.0015
2	10.0	0.050	10.0	0.10	0.0025
3	10.0	0.200	10.0	0.20	0.010
4*	10.0	0.500	10.0	0.50	0.025
5	10.0	1.000	10.0	1.00	0.050
6	10.0	1.200	10.0	1.20	0.060

7.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of Aroclors 1016/1260 at a

concentration of 0.5 ug/ml each time the initial calibration is performed. These are made by taking known aliquots of the ICV intermediate standard and diluting them to volume in hexane. If the analyst uses a project specified Aroclor in Section 7.4, then the analyst must verify that Aroclor with an ICV.

Table 2.2
ICV Working Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	10.0	0.500	10.0	0.500
Surrogate	0.5	0.500	10.0	0.025

7.6 Continuing Calibration Verification Standard (CCV): A working standard solution for Aroclor 1016/1260 at a concentration of 0.5 ug/ml is used to check the validity of a calibration curve on a daily basis. If the analyst uses a project specified Aroclor in Section 7.4, then the analyst must use that Aroclor as the CCV.

Table 2.3
CCV Working Standard

Working CCV Standard(s)	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	10.0	0.500	10.0	0.500
Surrogate	0.5	0.500	10.0	0.025

7.7 Surrogate standard: A commercially prepared certified solution of 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl is diluted in acetone to produce a working surrogate solution of 0.50 ug/mL. 0.5 mL is added to each sample and QC. The surrogate concentration is normalized to 100% from the spiking solution in the initial calibration. This will provide percent recoveries that transfer directly to LIMS.

Table 2.4
Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Surrogate	200	0.250	100.0	0.500

7.8 Spiking standards (matrix and control samples): Prepare a spiking solution in acetone that contains Aroclor 1016/1260 at a concentration of 10.0 ug/mL for water and sediment /soil samples. 0.5 ml is added to quality control and matrix spike samples. If client request a specified Aroclor, a spiking solution will be altered to match the Aroclor of interest

Table 2.5
Aroclor Spiking Solution

Aroclor Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	1000.0	0.500	50.0	10.00

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. The soil samples must be extracted within 14 days and water samples must be extracted in 7 days, from the date of collection.

8.2 Sample extracts are stored under refrigeration in the dark and analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

9.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in µg/L and the relative standard deviation (RSD) of the recovery Aroclors. In addition to each set of IDCs, a blind laboratory spike will be performed. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000, sec. 9.4).

9.3 Many programs (i.e. LCG, QSM) require the analysis of method reporting limit (MRL) standards and method detection limit (MDL) check standards as another means of checking method performance. The MRLs are analyzed at the beginning and end of each 12 hour shift and are typically prepared at concentrations equal to the reporting limits of the program. Recovery limits are program specific, but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x to 3x the method detection limit. MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). MDL check standards are derived from diluting the stock standard to a known concentration of 2x or 3x the method detection limit (Louisville Chemistry Guidelines, LCG). In most instances, a method detection limit check sample is analyzed at the beginning and end of each sequence requiring a MRL standards. If the MRLs fail for any compound, a MDL must be analyzed immediately following the failed MRLs. The recovery criteria for MDL check samples are the ability to detect all compounds at three times the noise level. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

9.4 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SURR, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Client and/or project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 3) also lists specific criteria to the method/project/program.

10.0 CALIBRATION

10.1 **Calibration**

10.1.1 The initial calibration for SW-846-8082 chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument across two dissimilar analytical columns. Each column must meet the initial calibration and continuing calibration criteria found in Table 3. In situations where only a few Aroclors are of interest for a specific project, the analyst will employ a multi-point initial calibration of each of the Aroclors of interest (e.g. five standards of Aroclor 1232 if this Aroclor is of concern and linear calibration models are employed)

and not use the 1016/1260 mixture or the pattern recognition standards.

10.1.2 Quantitation is based upon indicator peaks in the Aroclor 1016/1260 mixture that are generally present in the Aroclors. The calibration factors (CF), and generated Aroclors 1016/1260 from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation (SD), and the relative standard deviation (RSD) for each Aroclor peak. When the Aroclors 1016/1260 mixture is used to demonstrate detector response, the calibration model (linear or non-linear models) chosen for this mixture must be applied to the other Aroclors for which only standards are analyzed. A minimum of five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate the linearity.

10.1.3 The single point calibration standard will use a minimum of three to five peaks for each Aroclor. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of thee to five peaks are included and at least one peak that is unique to that Aroclor. The single standard for each of the other Aroclors will generate at least three calibration factors, one from each peak.

10.1.4 The identification of multi-peaked analytes such as PCBs as Aroclors can be performed by a combination of pattern recognition and retention time. Retention time windows are crucial to the identification of target compounds. Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in accordance with Method 8000 Section 11.6. Make three injections of target analyte standards over a course of 72-hour period. Each retention time must be to three decimal places. Calculate the mean and standard deviation of the target analytes. The width is 3x the standard deviation of the mean absolute retention time during the 72 hour period or 0.03 minutes, which ever is higher. If the standard deviation is 0.000, the laboratory will include more data points or use the default value of 0.01 minutes. Each analyte in each standard must fall within its respective retention time window. The retention time shall be set using the midpoint standard of the initial calibration curve or the value in the continuing calibration verification standard at the beginning of the analytical shift. If the analytes fall outside the established retention time window, the gas chromatographic

system must either be adjusted so that a second analysis of the standard results in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.1.5 Prepare the Aroclor 1016/1260 calibration standards (in table 2.1) at a minimum of five different concentrations by adding known volumes of the intermediate stock standard to a volumetric flask and diluting to volume with hexane. Note: QSM requires that the LOQ or the lowest point in the curve, which ever is greater, be used for the reporting limit.

10.1.6 The lowest concentration calibration standard that is analyzed during an initial calibration curve, establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

10.1.7 External standard calibration involves comparison of instrument response from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount of analyte in the calibration standard is defined as the calibration factor (CF). Aroclors are a multi-component standard. For the initial calibration curve, five peaks (or more) will be selected from each Aroclor 1016/1260 and calibrated using the average percent RSD of the peaks. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate the linearity.

10.1.8 Linear calibration using the average calibration factor. When calculating, both calibration factors and response factors are a measure of the slope of the calibration relationship and assume that the curve passed through the origin. If the measured relative standard deviation (RSD), is less than or equal to 20%, the use of a linear model is generally appropriate, and the calibration curve can be assumed to be linear and to pass through the origin.

10.1.9 Linear calibration using a least squared regression. When the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The approach is to employ a regression equation that does not pass through the origin. This approach can also be employed based on past experience of the instrument response.

10.1.10 Non-linear calibration. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable, and monotonic over the calibration range. The

statistical considerations in using a non-linear calibration model require more data than the traditional linear approaches. A quadratic (second order) model requires the use of six initial calibration points.

10.1.11 Directly inject the prepared calibration standards into the gas chromatograph. An external calibration technique is employed. One of the external standards will be at a concentration near, but above the method detection limit. If the RSD $\leq$ 20%, the correlation of r $\geq$0.995, or $r^2 \geq 0.990$ is obtained then the calibration is deemed acceptable.

10.1.12 Check the validity of the calibration by analyzing a ICV. The variance of any given compound shall not be more than +/- 15% difference. This standard must be run prior to sample analysis. If the percent drift or the percent difference criteria is not met, then the subsequent sample analysis for that analyte is not acceptable.

10.1.13 Continuous checking the validity of the calibration by analyzing CCVs. The variance of the Aroclor 1016/1260 mixture shall not be more than +/- 15% difference when compared to the mean calibration factor. The CCV must be injected at the beginning of the sequence, and at intervals of no less than one per 20 samples injections or twelve hours (after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded), and at the end of each sequence. The single-point standard for the other Aroclors are analyzed before the analysis of any samples, and will be analyzed after the analysis of the five 1016/1260 initial calibration in Section 10.1.2. Note: QSM samples must have CCVs injected every ten samples.

10.1.14 It is highly recommended to employ two standards (CCVs) at different concentrations to verify the calibration curve using non-linear calibration. One standard shall be near the quantitation limit or action limit. The choice of specific standards and concentrations is generally a method or project specific consideration (Method 8082, 7.6.6).

10.2 Calibration Calculations

10.2.1 When determining PCBs as Aroclors by the external standard technique, calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in Nanograms)}}$$

10.2.2 Five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture. Each peak actually has a ratio concentration of the final concentration of that Aroclor, method sums the calculated values of the five target peaks, thus generates the correct final concentration.

10.2.3 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation, and the relative standard deviation (RSD) for each Aroclor peak.

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (CF_I - \overline{CF})^2}{n-1}\right)}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

10.2.4 Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

10.2.5 The analyst will not force the line through the origin, but have the intercept calculated from the five data points. The use of linear

regression will not be used as a rationale for reporting results below the calibration range.

10.2.6 A linear least squares regression attempts to construct a linear equation of the form: $y= mx + b$, by minimizing the differences between the observed results(y_i, the response calculated from the constructed equation). The regression equation is:

$$y_i' = ax_i + b$$

where:

a = Regression coefficient or the slope of the line
b = The y-intercept
y_i' = Predicted (or calculated) response for the ith calibration standard.
x_i = Mass of analyte in the ith calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n} (y_i - y_i')^2$$

where n is the total number of calibration points. The regression calculations attempt to minimize the sum of squares, hence the name "least squares regression."

10.2.7 Weighting the sum of the squares of the differences will significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i \ (y_i - y_i')^2$$

where:

w_i = Weighting factor for the calibration standard (w=1 for unweighted least squares regression.
y_i = Observed instrument response (area or height) for the ith calibration standard.
y_i' = Predicted (or calculated) response for the ith calibration standard.
n = Total number of calibration standards.

10.2.8 Least Squares Equation (LSQ) weighting method to be used for calculation of least squares regression fits, either $1/x$ or $1/x^2$, gives increased importance to smaller concentrations and areas. LSQ weight can be applied to linear, quadratic, and cubic fits only.

10.2.9 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model will be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen will have no more than four parameters, i.e., if the model is polynomial, it will be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.2.10 The statistical considerations in developing a non-linear calibration model require more data that the more traditional linear approaches described above. Linear regression employ five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards.

10.2.11 The "goodness of fit" of the polynomial equation is evaluated by calculating the weighed coefficient of the determination (COD);

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

y_{obs} = Observed response (area) for each concentration of the calibration standard.

$\bar{y}$ = Mean observed response from the initial calibration

Y_I = Calculated response at each concentration from the initial calibrations.

n = Total number of calibration points (6 points for quadratic equation)

p = Number of adjustable parameters in the polynomial.

10.2.12 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0. In order to

be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99.

11.0 PREPARATION PROCEDURES

11.1 **Water Extraction (Method SW-846, 3535)**

11.1.1 Pre-rinse all glassware to be used in the extraction with methylene chloride (Pesticide Grade).

11.1.2 Mark the meniscus on the bottle for later determination of sample volume (Refer to section 11.1.21). If sample contains solid particulates, pour off water layer into a clean one liter amber jar, then mark the meniscus on the new sample jar.

11.1.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 1L of Milli-Q water to a one liter amber jar.

11.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and quantitatively transfer half of the sample into a one liter amber jar and label MS, then transfer the other half of the sample into another 1 liter amber jar, and label MSD. If there is no sample to perform a matrix spike/matrix spike duplicate, notify project management. The default sample QC is to perform a laboratory control spike duplicate.

11.1.5 Check and adjust the all samples extraction pH <2 with a 1:1 sulfuric acid solution. Stir sample until homogenized. Check pH.

11.1.6 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 0.5 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 0.5 mL of the Aroclor 1016/1260 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate). Stir sample.

11.1.7 Using a three place solid phase extraction unit (SPEU). Place the solid phase extraction apparatus (SPEA) on top of the SPEU. Remove SPEA funnel and place extraction disk on the stainless steel support screen. Replace reservoir funnel and clamp the reservoir in place on top of the disk.

11.1.8 Wash the disk with 10ml of methylene chloride.

11.1.9 Apply vacuum to dry to the disk.

11.1.10 Add 10 to 20mls of methanol to the SPEA. Apply vacuum and pull approximately 1 ml through the disk. Vent the vacuum and allow to disk to soak for 30 to 60 seconds.

11.1.11 Apply vacuum and pull methanol through the disk until the methanol surface is 3-5 mm above the disk surface.

11.1.12 Add 10 to 20mls of DI water to the SPEA, apply vacuum and pull the water through the disk until the water surface is 3-5 mm above the disk surface.

11.1.13 Pour the sample into the SPEA and apply vacuum. Recoveries are not affected by flow rate. Flow rate is dependent on vacuum source and particulate content of the sample. Flow rates through individual extraction disk may vary.

11.1.14 After sample extraction is complete, remove as much residual water as possible from the disk by applying vacuum to dry the disk for 5 minutes.

11.1.15 Remove the SPEA and place a clean 40ml VOA vial inside the SPEU. Replace the SPEA.

11.1.16 With 10mls of acetone, rinse down the sides of the sample container. Transfer the solvent from the sample container to the SPEA using a pipet, washing the walls of the SPEA in the process.

11.1.17 Apply vacuum and pull approximately 1ml of solvent through the disk. Vent to vacuum and allow the disk to soak for 30-60 seconds before applying vacuum to dry the disk.

11.1.18 Repeat steps 11.1.16 and 11.1.17.

11.1.19 Add 10mls of methylene chloride to the SPEA. Apply vacuum and pull approximately 1ml of solvent through the disk. Vent to vacuum and allow the disk to soak for 30-60 seconds before applying vacuum to dry the disk.

11.1.20 Repeat step 11.1.19.

11.1.21 Determine the sample volume by filling the sample bottle to the mark (11.1.2) with water and transferring it to a “Class A” 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet.

11.1.22 Samples can potentially require cleanup prior to concentration/analysis. Refer to attachments; I, II, III, and IV for sample cleanup procedure.

11.1.23 Refer to section 11.4 for sample concentration.

11.2 **Soil Extraction (Method SW-846, 3545)**

11.2.1 Preparing the extraction cell for use: Pre-rinse extraction cell (tube and caps) with methylene chloride. Attach the screw fit tube cap of the soil extractor to the end of the tube without the serial number engraved on the side. Using the filter rod, push 1 Dionex ASE filter through the open end of the tube until they reside flush on the bottom of the screwed end.

11.2.2 Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample more amenable to grinding.

11.2.4 Gummy, fibrous, or oily materials not amenable to grinding shall be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make sample easier to mix. Wipe samples can be placed directly into the cell.

11.2.5 (Refer to SOP FO-10 for subsampling guidance). Weigh approximately 10 g of sample (25g for PCB Low Level) to the nearest 0.1 g into a 250-mL beaker and record the final weight. Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples shall be a free flowing powder. If sample is not free flowing, add more diatomaceous earth until the sample has a dry texture. This powder is mixed so that the sample can pass through a 1 mm sieve

11.2.6 Transfer the ground sample to an extraction cell.

11.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10g of

sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

11.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh three individual 10gm grams aliquots of sample. Add 2.5gm drying agent. Transfer sample to extraction cell. If there is no sample available to perform a matrix spike/matrix spike duplicated, a laboratory control spike duplicate must be performed.

11.2.9 Fill the void in each of the extraction cells with clean sand. (Dionex ASE 200 Operator's Manual section 3.1.4 p.6).

11.2.10 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 0.5 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 0.5 mL of the Aroclor 1016/1260 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.2.11 Attach the other cap to the extractor cell, making sure the tube is sitting flush on a hard surface so that no particulates get caught in the threads of the tube cap.

11.2.12 Place the extractor tube on the Dionex ASE on a selected position with the serial number engraved end up on the top wheel. Place an appropriately labeled empty 60 mL VOA collection vial on the matching position on the bottom wheel. Schedule the Dionex ASE 200 and begin the cycle.

11.2.13 The Dionex ASE 200 extraction cycle:

Oven temperature: 100 º C Pressure: 1500 psi
Prepurge time: 0 minutes
Static time: 5 minutes
Heat: 5 minutes
Flush volume: 60%
Nitrogen purge: 60 sec. At 150 psi
Solvent A: 100%
Method rinse: ON
Static Cycles: 1
Extraction Fluid: (7:3) methylene chloride:acetone

11.2.14 Samples can potentially require cleanup prior to concentration and/or analysis. Refer to attachments; I, II, III, and IV for sample cleanup procedure.

11.2.15 Refer to section 11.4 for sample concentration.

11.3 **Waste Dilution Extraction (SW846-3580)**

11.3.1 (Refer to SOP FO-10 for subsampling guidance). Samples consisting of multiphase separations.

11.3.2 Pre-rinse "Class A" 10 ml volumetric with hexane.

11.3.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD).

11.3.4 Place the 10 ml volumetric on analytical balance (capable of accurately recording weight to the 0.01 g). Using a Pasteur pipet, transfer 1.0 g (to the nearest 0.1 g) to the volumetric. Record the weight.

11.3.5 Fill the volumetric half way with hexane.

11.3.6 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 0.5 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 0.5 mL of the Aroclor 1016/1260 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.3.7 Bring samples up to volume with hexane and cap for storage.

11.3.8 Add 2.0 grams of conditioned sodium sulfate to a 15ml amber vial with a Teflon cap. Transfer sample from the 10 ml volumetric flask to the 15ml vial.

11.3.9 Shake sample for two minutes.

11.3.10 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extracts through the glass wool and collect 5ml of the extract in a tube or vial.

11.3.11 No concentration step is need for this extraction. Sample can potentrially require cleanup prior to analysis. Refer to attachment I, II, III, and IV for sample cleanup options.

11.4 **Sample Concentration**

11.4.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds with sodium sulfate. Rinse filter paper, sodium sulfate, funnel, and TurboVap tube with methylene chloride.

11.4.2 Quantitatively pour the extract through the filter and funnel seated on a 200mL TurboVap concentrator tube (Kuderna-Danish (K-D) apparatus can be employed at this step). Rinse the beaker three times with methylene chloride. Add these rinses through the filter and funnel into the concentrator tube.

11.4.3 Before placing the concentrator tube into the unit, make sure that the water is at the appropriate level and temperature. Place the concentrator tube into the TurboVap unit and then lower gently so the tubes rest firmly on the sensor tray. The proper nitrogen pressure condition is set between 6-8 and the temperature 45ºC. It is critical that the analyst watch the extract as it concentrates. THE EXTRACT WILL NOT GO TO DRYNESS.

11.4.4 When the extract volume reaches approximately 4-6 mL, remove the concentrator tube from the bath and add 40 mLs of hexane. Use a pipet to aid in mixing of the solvents by drawing up the extract and expelling solvent, then gently stir the solvent to ensure that the methylene chloride and hexane are well mixed. Place the concentrator tube back into the unit. Concentrate the extract to approximately 5 mL. Remove the concentrator tube from the bath and allow the concentrator tube to cool completely.

11.4.5 With a disposable Pasteur pipet draw up the extract and rinse the sides of the concentrator tube. Repeat this rinsing several times. (Note: If the sample extract appears highly colored or has a strong petroleum type odor, a sulfuric acid clean-up will be required.) Transfer the extract from the concentrator tube using a Pasteur pipet to a 10mL volumetric flask. Rinse the concentrator tube with approximately 3mLs of hexane. Transfer the rinse to the volumetric flask. Bring sample up to a 10 mL volume with hexane. Transfer about 1 mL of the extract to a labeled screw-cap injection vial, and the remainder into an amber storage vial. Record the final extract on the injection extraction bench sheet.

11.4.6 The sample extract is now ready for analysis. If samples are not analyzed immediately store the sample extract in a refrigerator.

11.4.7 Samples can potentially require cleanup prior to analysis. Refer to attachment I, II, III, and IV for sample cleanup options.

12.0 SAMPLE ANALYSIS

12.1 **Sample Sequence**

12.1.1 Before initial calibration or sample analysis, a priming standard will be injected at a suggested level 2x the highest linearity point. A clean hexane blank must be analyzed to determine baseline characteristics. If the instrument has been in use within 24 hours prior to analysis, this step can be omitted.

12.1.2 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing Aroclors 1016/1260, prior to conducting any sample analysis. A CCV must be injected at the beginning of the sequence, and at intervals of no less than one per 20 samples injections or twelve hours (after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded), and at the end of each sequence. The variance of any given Aroclor 1016/1260 shall not be more than +/- 15% difference. If a different Aroclor is chosen for the continuing calibration verification, that CCV is subjected to the same QC criteria as the original CCV of Aroclors 1016/1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst can include a standard for one or all of these Aroclors after the 1016/1260 CCV for pattern reference.

12.1.3 Before the initial sample batch analysis, inject the sample prep batch method blank to demonstrate that the instrument, as well as the extraction procedure, is free from contamination. All compounds must be below the method detection limit.

12.1.4 Samples can be directly injected after the successful analyses of the calibration curve, ICV, CCV and method blank. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries will be within laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections.

12.1.5 All positive detects in the associated sample must be confirmed by a second column. Positive detects are identified and quantitated using both detectors. The results between primary and secondary columns must be within +/- 40 % difference, (if greater than 40% the target analyte is qualified with a “P”). If there is no evidence of chromatographic problems, then it is appropriate to report the lower

result (method 8000C). Specific projects or programs will require that the higher of the two columns results be reported.

12.1.5.1 QSM: Report the higher of two columns confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result. Qualify the data with a "Q" flag if target analyte is not confirmed by second column confirmation.

12.1.6 Each sample analysis must be bracketed with passing continuing calibration verification standards. If the CCV standard fails to meet QC criteria, all samples that were injected after the last standard which met QC criteria must be re-analyzed.

12.1.7 The data acquisition software generates a ug/ml concentrations which can then be applied to the final calculation to give sample concentrations. Samples with results exceeding calibration range are diluted accordingly in hexane and re-analyzed.

12.2 **Sample Calculations**

12.2.1 The concentration of each analyte in the sample is determined by the computer software. It calculates the amount of compound from the peak area, using the specified calibration model (CF, Linear Regression, or Quadratic). Each peak is a ratio concentration of the final concentration of that Aroclor, the method sums the calculated values of the five target peaks, thus generating the correct final concentration.

12.2.2 Calculation of the unknown sample by utilizing the CF is calculated as:

$$\text{Conc} = \frac{\text{Avg. Target Peak Areas}_{unk}}{\text{Avg. } \overline{CF}} \times MF$$

Conc. = the concentration of the unknown analytes of interest.
Amt_{unk} = the average sum of the uncorrected area of the unknown sample.
CF = Average sum of the Calibration Factors of the target peaks.
MF = multiplication factors (i.e. dilutions, percent moisture, etc.).

12.2.3 Using the slope (m) and the intercept (b) from this equation, the concentration of the sample (ug/ml) is calculated and appears on the report page following the chromatogram.

Water samples:

$$C_S = [(C_C)(V_E)(D)]/V_S$$

Soil samples:

$C_S=[(C_c)(V_E)(D)]/[(W)(S)]$

Wipe samples:

$C=[(C_c)(V_E)(D)]$

Where: C_S = Concentration of sample in ug/L for waters and mg/kg on a dry weight basis for soils

C_c = Concentration from the curve (ug/ml)

C = Concentration in total ug

V_E = Total volume of sample extract (after concentration) in ml
V_S= Volume of water sample in liters

D = Dilution factor if extract was diluted
W = Weight of wet soil sample in grams

S = Total percent solids, expressed as: percent total solids/100

13.0 DATA ASSESSEMENT & ACCEPTANCE CRITERIA FOR QC MEASURES

13.1 **Data Assessment & Acceptance Criteria for QC Measures**

13.1.1 If the initial analysis of a sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be re-analyzed at a dilution.

13.1.2 The qualitative identification of PCBs as Aroclors determined by this method is based on retention time and pattern recognition. This method employs a dual ECD detector with two dissimilar columns. There is a potential for many non-target compounds present in samples to interfere with this analysis, therefore sample extracts can undergo cleanup procedures. The retention times are updated at the midpoint of each new calibration and they are continually updated with the daily continuing calibration verification standards (CCVs). A new method ID is provided for each new sequence with updated retention times or alteration to the operating initial calibration method. If an alteration occurs (other than daily retention time updates), project management and the QA officer will be notified. A description of the alteration will addressed in the client notes or case narrative.

13.2 **Reporting Quantitative Analysis**

13.2.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times are compared with the sample retention times, the sample data can be reported. Assessments of all spiked control samples, and standards will be scrutinized before reporting the data.

13.2.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors, and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 4.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs his or her portion of the checklist (the checklist is included in this SOP). Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

13.2.3 An electronic copy of the data is then filed and archived. The package includes; the sequence run log, a copy of the bench sheet, the LIMS run log, LIMS prep sheet, verification of calibration data, each sample's chromatogram. All the data is e initialed and dated by the analyst. Each sequence file header is labeled with the date of sequence.

13.3 **Corrective Measures for Out-of-Control Data**

13.3.1 When data is out of control, corrective actions are required. Certain situations will cause data deficiencies. If the non-conformities involve failing QC within the analytical sequence batch, the reanalysis of samples will eliminate any out of control data. If the out of control data is the result of instrument, malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data shall correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument will need cleaning and decontamination. The sample will need diluting or undergo sample clean-up procedures to reduce matrix affect. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions shall be taken for different QC-type failures or non-conformities (Table 3).

13.3.2 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data can be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.

13.3.3 To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises re-analysis or re-extraction is an option.

13.3.4 If a sample is extracted passed holding times, the analyst must notify client services immediately. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur. If the client still request sample analysis, the sample data is qualified for holding time violations.

13.3.5 If the non-conformities involve failing QC within the analytical sequence batch, the analyst must use professional judgment for the reanalysis of the extracts. The reanalysis will ensure that the instrument was functioning properly. If the results are passing, the non-conformities were associated with the instrument. If data is still outside QC criteria, re-extraction of the all samples associated with the QC samples must be performed (provide that there is sample available). If no sample is available for re-extraction, the samples are qualified accordingly.

13.3.6 If the out of control data is the result of instrument malfunctions, the analyst must evaluate and repair the instrument prior to reanalysis of the affected data. After the instrument maintenance and repair the corrective measures must be demonstrated by analyzing a Continuing Calibration Verification (CCV) before continuing sample analysis. Any maintenance and repair must be noted in the instrument logbook.

13.3.7 If sample matrix affect or contamination is the reason for poor data, the analyst will use professional judgement for the reanalysis of the extract. The extract can be diluted and reanalyzed. The sample can be re-extracted confirming matrix interference. The analyst can report and qualify the sample data. The analyst must supply explanation for the qualified data in the narrative or client notes.

13.3.8 If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding

and/or correcting those errors. Senior analysts have the authority to validate their own data.

13.3.9 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

13.3.9.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

13.3.9.2 In all instances, if results are reported from data that is out of control or unacceptable, that data is qualified accordingly. Once the client has been notified and he or she instructs us to report the data, data is qualified indicating what type of nonconformity has occurred.

13.3.9.3 Out of control data is still retained by the laboratory, filed and archived along with acceptable data.

13.3.9.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating the nonconformance and to ensure that the proper corrective actions are employed.

14.0 WASTE MANAGEMENT

14.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

15.0 REFERENCES

15.1 USEPA, SW-846, 3rd Ed. Chapters 2&4, Method 8000C. March 2003.

15.2 USEPA, SW-846, "Polychorinated Biphenyls (PCBs) by Gas Chromatography", Method 8082 Revision 0. December 1996.

15.3 USEPA, SW-846, "Organic Extraction and Sample Preparation", Method 3500B Revision 2. December 1996.

15.4 USEPA, SW-846, "Solid Phase Extraction (SPE)", Method 3535 Revision 0. December 1996.

15.5 USEPA, SW-846, "Pressurized Fluid Extraction", Method 3545 Revision 0. December 1996.

15.6 Department of Defense, "Quality Systems Manual for Environmental Laboratories". Version 3. January 2006.

15.7 3M Empore Extraction Disk for Environmental Analysis.

ATTACHMENT I
SULFUR CLEANUP
METHOD 3660

1.0 SCOPE AND APPLICATION

This method covers the procedures used to eliminate sulfur interference from PCB extracts. Elemental sulfur is indicated by the presence of white crystals in the sample extract, or, upon analysis, a broadband interference from the solvent front.

2.0 REFERENCES

USEPA, SW-846, 3rd Ed. Method 3660. December, 1996.

3.0 APPARATUS AND MATERIALS

3.1 Mechanical shaker or mixer

3.2 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

3.3 Disposable culture tubes – 25 mL with Teflon-lined screwcaps.

3.4 Tetrabutylammonium (TBA) – sulfite reagent.

3.5 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212).

4.0 REAGENTS

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Organic-free reagent water.

4.3 Acetone, pesticide grade.

4.4 Hexane, pesticide grade.

4.5 2-Propanol, pesticide grade.

4.6 Tetrabutylammonium (TBA) sulfite reagent. (Aldrich Part # 86847-1EA-F).

4.7 Tetrabutylammonium hydrogensulfate (Aldrich Part # 155837-25G).

4.8 Sodium sulfite, (Aldrich Part# 239321-500G).

4.9 Prepare reagent by dissolving 3.39g tetrabutylammonium hydrogen sulfate in 100 ml organic-free water. To remove impurities, extract this solution 3 times with 20 ml portions of hexane. Discard the hexane extracts, and 25g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a PTFE-lined screw cap. This solution can be stored at room temperature for at least one month.

4.10 Nitric Acid, HNO_3, dilute

4.11 Copper powder, (UCT Part# ECCU01K)

5.0 PROCEDURE

5.1 TBA Solution

5.1.1 Pipet 1.0 mL of PCB extract into a 25 mL culture tube.

5.1.2 Add 1.0 mL TBA – sulfite reagent and 2 mL iso-propanol to the tube and cap.

5.1.3 Shake for at least 1 minute. If the sample is colorless or if the initial color is unchanged, and if crystals are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100 mg portions until a solid residue remains after repeated shaking.

5.1.4 Add 5 mL distilled water and shake for at least 5 minutes. Allow the sample to stand for 5-10 minutes. Transfer the hexane layer to injection vials and/or storage vials.

5.1.5 Analyze the extracts by gas chromatography.

5.1.6 Verify that the TBA-sulfite is free from contamination by shaking out 1.0 mL of TBA – sulfite with 10 mL of hexane and analyze.

5.1.7 Process all quality control samples (e.g., spikes, blanks and duplicates) along with any samples.

5.1.8 Verify that recoveries of PCB's are greater that 80% by processing a standard (CCV) through the procedure.

5.2 Copper Powder

5.2.1 Remove oxides by treating the copper powder with dilute nitric acid, rinse with organic free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

5.2.2 Concentrate the sample to exactly 1.0ml or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

5.2.3 If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the concentration tube. Transfer 1.0ml of the extract to the calibrated centrifuge tube.

5.2.4 Add approximately 2g of cleaned copper powder to the centrifuge tube. Vigorously mix the extract and the copper powder for a t least 1 min on the mechanical shaker. Allow phases to separate.

5.2.5 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1 ml of extract. This step is necessary to prevent further degradation of the pesticides.

ATTACHMENT II
FLORISIL CARTRIDGE CLEANUP
METHOD 3620

1.0 SCOPE AND APPLICATION

1.1 Florisil, a registered trade name of U. S. Silica Co., is a magnesium silicate with basic properties. It is used to separate analytes from interfering compounds prior to sample analysis by a chromatographic method.

1.2 Florisil has been used for the cleanup of pesticide residues and other chlorinated hydrocarbons; the separation of nitrogen compounds from hydrocarbons; the separation of aromatic compounds from aliphatic-aromatic mixtures; and similar applications for use with fats, oils, and waxes. Additionally, Florisil is considered good for separations of steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates. Florisil cleanup is accomplished by using a glass chromatographic column packed with Florisil or using solid-phase extraction cartridges containing Florisil. This method includes procedures for cleanup of sample extracts containing the following analyte groups:

Chlorinated hydrocarbons
Organochlorine pesticides
Organophosphates
Organophosphorus pesticides
PCBs
Phthalate esters
Nitrosamines
Nitroaromatics
Haloethers
Aniline and aniline derivatives

2.0 SUMMARY OF METHOD

1.1 This method describes procedures for Florisil cleanup of solvent extracts of environmental samples using solid-phase extraction cartridges. The cartridge cleanup protocol uses solid-phase extraction cartridges containing 40 µm particles of Florisil (60 D pores). Each cartridge is washed with solvent immediately prior to use. The sample extract is loaded onto the cartridge which is then eluted with suitable solvent(s). A vacuum manifold is required to obtain reproducible results. The eluate will be re-concentrated prior to gas chromatographic analysis.

2.0 INTERFERENCES

2.1 A reagent blank will be prepared and analyzed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples. The procedures for reagent purification outlined here will be considered to be the minimum requirements for use of this method. More extensive procedures will be necessary to achieve acceptable levels of interferences for some analytes. However, during the evaluation of the cartridge clean-up procedure, phthalate esters were detected in the Florisil cartridge method blanks at concentrations up to 400 ng per cartridge. Therefore, complete removal of the phthalates esters from Florisil cartridges are not possible.

3.0 APPARATUS AND MATERIALS

3.1 Vacuum manifold - VacElute Manifold SPS-24 (Analytichem International), Visiprep (Supelco, Inc.) or equivalent, consisting of glass vacuum basin, collection rack and funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500-mL sidearm flask fitted with a one-hole stopper and glass tubing. The manifold is required for use of the cartridge cleanup protocol.

4.0 REAGENTS

4.1 Eluting solvents - all solvents must be pesticide quality or equivalent.

4.2 Hexane, pesticide grade.

4.3 Methylene chloride, pesticide grade.

4.4 Acetone, pesticide grade.

4.5 Florisil cartridge phenol check solution (for the organochlorine pesticide technique) - Prepare a solution of 2,4,5-Trichlorophenol in acetone at a concentration of 0.1 g/L.

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4.6 Florisil cartridge pesticide check solution - Prepare a solution containing the following analytes in hexane:

BHCs
Heptachlor
BHC
Endosulfan I
Dieldrin
Endrin
4,4'-DDD
4,4'-DDT
Methoxychlor
Tetrachloro-m-xylene
Decachlorobiphenyl

5.0 PROCEDURE

5.1.1 Whenever Florisil is used to fractionate groups of target compounds (rather than to simply remove potential interference) it is critical that the specific fractionation scheme be validated using spiked solutions or spiked sample extracts that contain most or all of the analytes of interest. This is particularly important when the Florisil cartridge techniques are employed, as the differences between the various cartridge formats and manufacturers will affect the fractionation patterns. In addition, it will be useful to archive any fractions not originally intended for analysis, in the event that the fractionation scheme chosen does not yield the intended results. Once the determinative analysis has been performed and demonstrates that the fractionation has been successful, such archived fractions will be disposed of in an appropriate manner. However, if the fractionation did not perform as intended, the analytes of interest contained in the archived fractions, which will be able to be analyzed or combined with the other fraction(s) for reanalysis.

5.1.2 Following Florisil cleanup, extracts will require further concentration and/or solvent exchange.

5.2 Cartridge Evaluation

5.2.1 The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are to be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

5.2.2 To check each new lot of Florisil cartridges before use, perform the following in duplicate.

5.2.3 Combine 0.5 ml of 2,4,5-Trichlorophenol solution in Section 5.5 and 0.5 ml of pesticide spiking solution in Section 5.6 in a two ml autosampler vial.

5.2.4 Arrange the cartridges on the manifold in the closed-valve position.

5.2.5 Turn on the vacuum pump and set the vacuum to 10 in (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates are controlled by opening and closing cartridge valves.

5.2.6 Condition the cartridges by adding 4 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

5.2.7 Slowly open cartridge valves to allow the hexane to pass through the cartridges.

5.2.8 Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed.

5.2.9 Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

5.2.10 Add the spiking solution in 6.2.3 to the Florisil cartridge.

5.2.11 Elute the cartridge with 9 ml of acetone/hexane (10/90, v/v). Collect the extraction in a 15 ml test tube.

5.2.12 Transfer extract to a concentrating thimble. Using the nitrogen blow down apparatus, concentration the extract down to one ml and analyze by Method 8081A.

5.2.13 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110%, if the trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

5.3 Handling sample extracts

5.3.1 Most sample extracts will have to be concentrated to a smaller volume prior to the use of Florisil cleanup. The extract volume is a function of the analytical sensitivity necessary to meet the project objectives. The extract volume will also affect the ability of the Florisil to separate target analytes from potential interferences where applying large extract volumes to the cartridges will cause poor results. Consult the appropriate extraction and determinative methods for the details on final extract volumes, extract concentration techniques, and solvent exchange procedures.

5.3.2 Reduce the sample extract volume to 10 mL prior to cleanup. The extract solvent will be hexane for these analytes. In most cases, given the sensitivity of the determinative methods, only 1 mL of the 10 mL extract needs to be subjected to the Florisil cleanup procedure. The remaining 9 mL will be archived for later use, if needed.

5.3.3 Place Florisil cartridges on manifold. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Condition the cartridges by adding 4 ml of hexane. Slowly open the cartridge valve and collect the eluate into the collection vial. Allow a few drops of hexane to pass through the cartridge to remove any air bubbles. Close valve and allow solvent to soak the entire sorbent bed for five minutes. Do not turn off vacuum.

5.3.4 Slowly, open the valve to allow the hexane to pass through the cartridge. Close valve when about 1 mm of hexane is remaining above the sorbent bed.

5.3.5 Add 1 ml of extract to the Florisil cartridge. Allow the extract to pass through the cartridge at a rate about 2 ml per minute.

5.3.6 Before extract goes below the surface of the sorbent bed, add 9 ml of hexane/acetone (90/10, v/v) to the cartridge.

5.3.7 After extract has been collected, concentrate sample down to 1 ml. Refer to Section 9.5 for concentration techniques.

The following procedures are used to separate the organochlorine pesticides from the PCBs.

5.3.8 Add 3 mL of hexane to the cartridge. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Allow the solvent to soak the sorbent bed for 1 minute or less. Slowly open the cartridge valve and collect the eluate into the collection

vial. This is Fraction 1 and it will contain the PCBs and a few of the organochlorine pesticides.

5.3.9 Close the cartridge valve, replace the collection vial, and add 5 mL of methylene chloride/hexane (26/74, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 2 and it will contain most of the pesticides.

5.3.10 Close the cartridge valve, replace collection vials, and add 5 mL of acetone/hexane (10/90, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 3 and it will contain the remaining pesticides.

5.3.11 As needed, perform a solvent exchange and adjust the final volume of the eluant to 1 ml.

6.0 QUALITY CONTROL

6.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentration. The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are to be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

6.2 Organochlorine pesticides - To check each new lot of Florisil cartridges before use, perform the following in duplicate. Combine 0.5 mL of the 2,4,5-trichlorophenol solution, 1.0 mL of the pesticide solution, and 0.5 mL of hexane in a vial. Condition the cartridge and then perform the cartridge cleanup Elute the cartridge with 9 mL of acetone/hexane (10/90, v/v) only. Reduce the volume to 1.0 mL and analyze. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110 %, if the recovery of trichlorophenol is less than 5 %, and if no peaks interfering with the target analytes are detected.

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ATTACHMENT III
SULFURIC ACID CLEANUP
METHOD 3665

1.0 SCOPE AND APPLICATION

1.1 This method is suitable for the rigorous cleanup of sample extracts prior to analysis for PCBs. This method will be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs. This method cannot be used to cleanup extracts for other target analytes, as it will destroy most organic chemicals including pesticides.

1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 An extract is solvent exchanged to hexane, then the hexane is sequentially treated with concentrated sulfuric acid.

2.2 Blanks and replicate analysis samples must be subjected to the same cleanup as the samples associated with them.

3.0 INTERFERENCES

3.1 This technique will not destroy chlorinated benzenes, chlorinated naphthalenes, and a number of chlorinated pesticides.

4.0 APPARATUS

4.1 Syringe or Class A volumetric pipet, glass; 1.0, 2.0, and 5.0 ml.

4.2 Vials – 1, 2, and 10 ml, glass with polytetrafluoroethylene (PTFE)-lined screw caps.

4.3 Vortex mixer

5.0 REAGENTS

5.1 Organic-free water (MilliQ-processed)

5.2 Sulfuric acid/water, H_2SO_4/H_2O (1:1, v/v)

5.3 Hexane, pesticide grade

6.0 PROCEDURE

6.1 Using a syringe or a volumetric pipet, transfer 1.0 or 2.0 ml of the hexane extract to a 10 ml vial and carefully add 5 ml of the 1:1 sulfuric acid/water solution. Mix well.

6.2 Allow the phased to separate for at least 1 minute. Examine the top hexane layer, a clear or slightly tinted layer is present, it will not be highly colored nor will it have a visible emulsion or cloudiness. If clean hexane phase is not achieved repeat step one.

6.3 Transfer the hexane layer to a clean 5 ml vial.. Take care not to include any of the acid layer in this clean vial. Once the hexane layer is removed, perform a second "extraction" of the acid layer.

6.4 Add an additional 1 ml of hexane to the sulfuric acid layer, cap and mix well. This second extraction is done to ensure quantitative transfer of the PCBs or Toxaphene.

6.5 Remove the second hexane layer and combine with the clean extract

6.6 Reduce the volume of the combined hexane layers to the original volume (1 or 2 ml) using an appropriate concentration technique.

6.7 The extracts obtained are now ready to be analyzed for the target analytes using the appropriate organic techniques. If analysis of the extract will not be performed immediately, transfer extract to a vial with a PTFE-line screw cap top.

ATTACHMENT IV
GEL-PERMEATION CLEANUP
METHOD 3640A

1.0 SCOPE AND APPLICATION

1.1 Gel-Permeation Chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinylbenzene-styrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

1.2 General cleanup application – GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes; therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

1.3 Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue will ultimately reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem. GPC, operating on the principal of size exclusion, will not usually remove interference peaks that appear in the chromatogram since the molecular size of these compounds is relative similar to the target analytes. Separation cleanup techniques, based on other molecular characteristics (i.e. polarity), must be used to eliminate this type of interference.

1.4 This method is restricted for use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method is used to cleanup extracted samples with unwanted light and/or heavy compounds that interfere with final analysis of methods; 8081, 8082, 8270 and 8310.

2.2 Samples are extracted then concentrated and prepared for GPC cleanup.

2.3 Determinations for which samples that are candidates for GPC cleanup are based on client specific, site specific and project specific requirements, specific characteristics of initial extract (e.g. color or odor) or interference determined by initial extract analysis.

2.4 The column is calibrated and then loaded with the sample extract to be cleaned up. Elution is effected with a suitable solvent(s) and collection times are adjusted based on desired analysis. The collected fractions are then concentrated per guidelines for methods; 8081, 8082, 8270 and 8310.

3.0 INTERFERENCES

3.1 A reagent blank is to be analyzed for the compound of interest prior to the use of this method. The level of interferences must be below the estimated quantitation limits (EQL's) of the analytes of interest before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method will be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Gel-permeation chromatography system - Gilson GV-271 ASPEC, Gilson, INC or equivalent. All systems, whether automated or manual must meet the calibration requirements.

4.2 Chromatographic column -350mm x 21.20 mm 0 micron, Phenomenex p/n 00W-3035-PO or equivalent.

4.3 Guard column – (optional) 60mm x 21.20 mm 0 micron, Phenomenex p/n 03R-3035-PO or equivalent.

4.4 Ultraviolet detector –fixed wavelength (254 nm) with a semi-prep flow-through cell, Gilson 112 UV/VIS detector, Gilson, INC..

4.5 Strip chart recorder, recording integrator or laboratory data system, (Trilution and LIMS).

4.6 Syringe – 10ml with Luerlok fitting.

4.7 Syringe filter assembly, disposable – Puradisc 25TF sample filter assembly Whatman #6784-2510, 25mm and 1.0 micron filter discs or

equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

4.8 Analytical balance – 0.00 g.

4.9 Volumetric flasks, Class A 5ml to 100ml

4.10 Graduated cylinders

4.11 Disposable glass culture tubes 13mm x 100mm, (Kimble 73500-13100).

4.12 Disposable glass collection tubes- (Kimax 51 25mm x 200mm, Kimble 45060-25200).

4.13 Pasteur pipets- (Corning #7095B-5X)

Reagents

4.14 Methylene chloride, Pesticide grade.

4.15 Cyclohexane, Pesticide grade.

4.16 N-Butyl chloride, Pesticide grade.

5.0 CALIBRATION

5.1 Preparation of standards is documented in the GPC standard logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards once opened expire within six months or sooner if routine QC indicates a problem and not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire in six months and not to exceed the opened date of the stock standard and not to exceed manufactures expiration date.

5.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Stock standards once opened expire in six months and not to exceed the manufactures expiration date.

5.3 The following is the stock standard that is commercially prepared standard, which is certified by the manufacturer;

GPC Calibration Mix: Restek Part # 32019. (1ml ampul)
GPC Calibration Mix: Restek Part # 32023. (5ml ampul)

5.4 GPC Calibration Solution is a certified prepared standard in methylene chloride containing the following analytes (in elution order):

Compound	mg/mL
Corn oil	250
Bis (2-ethylhexyl) phthalate	10
Methoxychlor	2.0
Perylene	0.2
Sulfur	0.8

Table 1.0 GPC Calibration Standard

Standard	Component Name	Conc. (mg/ml)	STD Volume (ml)	Final Volume (ml)	Final Conc. (ug/ml)
GPC STD	Corn Oil	250	5.0	50.0	25000.0
	Bis(2-ethylhexyl)pht.	10	5.0	50.0	1000.0
	Methoxychlor	2.0	5.0	50.0	200.0
	Perylene	0.2	5.0	50.0	20.0
	Sulfur	0.8	5.0	50.0	80.0

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND EXTRACTION PROCEDURES

6.1 Follow guidelines listed in section 8.0 in SOP 8081 Rev. 8 for sample collection, preservation and handling.

7.0 PROCEDURE

7.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 72°F.

7.2 GPC Setup

Column Preparation:

7.2.1 Using manual control options in Trilution set the system to equilibrate for at least 30 minutes prior to starting a run or priming.

7.2.2 If system has not been used on a regular basis, prime all lines and pumps either manually or using the automated system following guidelines listed in the Trilution help manual.

7.3 Calibration of the GPC Column

7.3.1 Place approximately 6 to 7 mls of the calibration solution in a disposable culture tube. Place tube in position 1 on the sample tray. Set Trilution to run a calibration method with an inject volume of 5.50ml.

7.3.2 Following are criteria for evaluation of the UV chromatogram for column condition.

7.3.3 Peaks must be observed and will be symmetrical for all compounds in the calibration solution.

7.3.4 Corn oil and phthalate peaks must exhibit >85% resolution.

7.3.5 Phthalate and methoxychlor peaks must exhibit >85% resolution.

7.3.6 Methoxychlor and perylene peaks must exhibit >85% resolution.

7.3.7 Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

7.3.8 Nitroaromatic compounds are particularly prone to adsorption. For example 4-nitrophenol recoveries can be low due to a portion of the analyte being discarded after the end of collection time. Columns are to be tested with the semivolatiles matrix spiking solution. GPC elution will continue until after perylene has eluted or long enough to recover at least 85% of the analytes, whichever time is longer.

7.3.9 Calibration for Semivolatiles – Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the

elution of perylene. Collection can be stopped before sulfur elutes. Each laboratory is required to establish its specific time sequences.

7.3.10 Calibration for Organochlorine Pesticides/PCBs – Determining the elution times for the phthalate, methoxychlor, perylene and sulfur. Choose a dump time which removes >85% of the phthalate, but collects >95% of the methoxychlor. Stop collection after the elution of perylene, but before sulfur elutes.

7.3.11 Calibration for Polynuclear Aromatic Hydrocarbons – Determine the elution times for corn oil, phthalate and perylene. Start elution collections just before the elution of bis (2-ethylhexyl) phthalate and end collection after the elution of perylene.

7.3.12 Verify the flow rate by collection column eluate for 10 minutes in a graduated cylinder and measure the volume which will between 45-55ml (4.5-5.5 ml/min). If flow rate is outside of this range, corrective action must be taken, as described above. Once flow rate is within the ranges of 4.5-5.5 ml/min, record the column pressure (6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If flow rate and/or column pressure do not fall within the above ranges the column or pre-column are suspected. A UV trace that does not meet the criteria in section 7.2.2.2 would also indicate that the column or pre-column are responsible the poor performance.

7.3.13 Reinject the calibration solution after appropriate collect and dump cycles have been set and the solvent flow and column pressure have been established.

7.3.14 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed can be used to indicate problems with the system during sample processing.

7.3.15 The retention times for bis (2-ethylhexyl) phthalate and perylene must not vary more than ± 5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by:

7.3.15.1 Poor laboratory temperature control or system leaks.

7.3.15.2 An unstabilized column that requires pumping methylene chloride through it for several more hours.

7.3.15.3 Excessive laboratory temperatures, causing outgassing of the methylene chloride.

7.3.16 Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by whatever detectors will be used for the analysis of future samples. Exchange the solvent if necessary. If the blank exceeds the estimated quantitation limit of the analytes, pump additional methylene chloride through the system for 1 to 2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

7.3.17 A hard copy calibration report will follow the samples in the sample prep folder.

8.0 EXTRACT PREPARATION

8.1 Adjust the extract volume to 5 ml. The solvent extract must be primarily methylene chloride. All other solvents, e.g. 1:1 methylene chloride/acetone, must be concentrated to 1 ml (or as low as possible if a precipitate forms) and diluted to 5 ml with methylene chloride. Thoroughly mix the extract before proceeding.

8.2 Filter the extract through a 1 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 ml syringe. Draw the sample extract through the filter assembly and into the 10 ml syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container, e.g. a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into a syringe without the filter assembly, attach the filter assembly and force the extract through the filter and into the glass container. The latter is the preferred technique for viscous extracts or extracts with a lot of solids. Particulate larger than 5 microns can scratch the valve, which can result in a system leak and cross-contamination of sample extracts in the sample loops.

NOTE**:** Viscosity of a sample extract can not exceed the viscosity of 1:1 water/glycerol. Dilute samples that exceed the viscosity.

9.0 SCREENING THE EXTRACT

9.1 Screen the extract to determine the weight of dissolved residue by evaporating a 100 µL aliquot to dryness and weighing the residue. The weight of dissolved residue loaded on the GPC column cannot exceed 0.500 g. Residues exceeding 0.500 g will very likely result in incomplete

extract cleanup and contamination of the GPC switching valve (which results in cross-contamination of sample extracts).

9.1.1 Transfer 100µL of the filtered extract from section 8.2 to a tared aluminum weighing dish.

9.1.2 A suggested evaporation technique is to use a heat lamp. Set up a 250 watt heat lamp in a hood so that it is 8 ± 0.5cm from a surface covered with a clean sheet of aluminum foil. Surface temperature will be between 80-100°C (check temperature by placing a thermometer on the foil and under the lamp). Place the weighing dish under the lamp using tongs. Allow it to stay under the lamp for 1 minute. Transfer the weighing dish to an analytical balance or a micro balance and weigh to the nearest 0.1mg. If the residue weight is less than 10mg/100µL, then further weighings are not necessary. If the residue weight is greater than 10mg/100µL then determine if constant weight has been achieved by placing the weighing dish and residue back under the heat lamp for 2 or more additional 0.5 minute intervals. Reweigh after each interval. Constant weight is achieved when three weights agree within ±10%.

9.1.3 Repeat the above residue analysis on a blank and a spike. Add 100µL of the same methylene chloride used for the sample extraction to a weighing dish and determine residue as above. Add 100µL of a corn oil spike (5g/100ml) to another weighing dish and repeat the residue determination.

9.1.4 A residue weight of 10mg/100µL of extract represents 500mg in 5ml of extract. Any sample extracts that exceed the 10mg/100µL residue weight must be diluted so that the 5ml loaded on the GPC column does not exceed 0.500g. Following is a calculation that will be used to determine what dilution is necessary if the residue exceeds 10mg.

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{10\text{mg maximum}}{\text{X mg of residue}}$$

Example

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{10\text{mg maximum}}{15 \text{ mg of residue}}$$

Y ml taken for dilution = 3.3 ml

Therefore, taking 3.3 ml of sample extract from 7.4.2 and diluting it to 5ml with methylene chloride will result in 5 ml of diluted extract loaded on the GPC column that contains 0.500 g of residue.

NOTE**:** This dilution factor must be included in the final calculation of analyte concentrations.

10.0 GPC CLEANUP

10.1 Calibrate the GPC at least once per week following the procedure outlined in section 7.3. Ensure that UV trace requirements, flow rate and column pressure criteria are acceptable. Also, retention time shift must be <5% when compared to retention times in the last calibration UV trace.

10.2 If these criteria are not met, follow appropriate maintenance methods to try to regain resolution.

10.3 Load sample vials with 5 ml of filtered extract from section 7.4.2. The Method Blank (MB) and Laboratory Control Standard (LCS) must be run through the GPC process from each prep batch that the samples originated from.

NOTE**:** The number of samples from each prep batch that are put through GPC will vary by client, location and project specifications

10.4 Set the GPC up to run cleanup of the samples, verifying that collection and dump times correlate with the calibration times for each analysis specified in section 7.3. Multiple methods can be used on each GPC run. Follow software specific guidelines for setting up a GPC run.

NOTE**:** It will be necessary to run multiple rinse methods between samples to ensure no cross-contamination between particularly dirty samples

10.5 Monitor sample volumes collected. Changes in sample volumes collected will indicate one or more of the following problems:

10.5.1 Change in solvent flow rate caused by channeling in column or changes in column pressure.

10.5.2 Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

10.5.3 Leaks in the system or significant variances in room temperature.

10.6 Concentrate the extract by following the methods listed in the sections for sample concentration for methods; 8081, 8082, 8270 and 8310.

11.0 QUALITY CONTROL

11.1 The analyst will demonstrate that the compound(s) of interest are being quantitatively recovered before applying this method to actual samples.

11.2 For sample extracts that are cleaned up using this method, the associated quality control samples must also be processed through this clean up method.

11.3 Some brands of methylene chloride can contain unacceptably high levels of acid (HCl). Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer.

11.4 If the pH of the water layer is ≤5, filter the entire supply of solvent through a 2 in x 15 in glass column containing activated basic alumina. This column will be sufficient for processing approximately 20-30 liters of solvent. Alternatively, find a different supply of methylene chloride.

Table 3
Summary of Method Objectives for Method 8082-PCBs by Aroclors

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Initial Calibration Aroclors 1016/1260 or Client/site specified Aroclor(s)	Initial Calibration prior to sample analysis. 1. RSD for each Aroclor $\leq$ 20% 2. Linear – least squares regression $r \geq 0.995$. 3. Non-linear regression $r^2 \geq 0.990$. (6 points shall be used for second order)	$r \geq 0.995$, RSD $\leq$20%, $r^2 \geq$0.990, mean RSD $\leq$ 20% QSM: $r \geq 0.995$ RSD $\leq$ 20%	For Aroclor analysis, a mixture of Aroclors 1016/1260 is normally used to establish detector calibration linearity, unless project specific Aroclor(s) is required. Linearity must fit one of acceptance criteria, or the curve must be reanalyzed. Single level CFs for the remaining Aroclors must be established with each initial calibration.
Initial Calibration Verification (ICV)	After initial calibration	85-115% QSM: 80-120%	If ICV falls outside QC criteria, reanalysis must take place. If ICV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". QSM: No samples will be analyzed until the problem has been corrected.
Continuing Calibration Verification (CCV)	Every twelve hours or every twenty samples injections (after10 sample injections is recommend) and at the end of the sequence. QSM: Every 10 samples	D or mean D$\leq$15% with a maximum %D$\leq$30% QSM: 80-120%	If CCV falls outside QC criteria, reanalysis must take place. If CCV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". Exception to the above, if the acceptance limits are exceeded high and the analyte is not detected in the sample the verification standard has passed (analyte would have been detected if present). QSM: No samples will be analyzed until the problem has been corrected.
Retention Time Window (RTW)	Retention Times will be set using the midpoint standard in the ICAL or the RT in the CCV run at the beginning of each analytical shift.	Retention times are 3x Standard Deviation the absolute retention time, if the default standard deviation is employed, width of the window will be 0.03 minutes	QSM: No samples shall be run without a verified retention time window at the initial verification. For CCVs, apply a "Q" to all results for analytes outside the retention time window.
MDL Study/MDL Verification Check	At initial set-up and annually for MDL Study. MDL Verification Checks shall be performed quarterly.	MDL checks must produce a signal at least 3 times the instrument's noise level.	Samples can not be analyzed without a valid MDL. QSM: Run MDL verification check at higher (2x MDL).

Table 3
Summary of Method Objectives for Method 8082-PCBs by Aroclors

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
MRL Level Verification Check standard at Reporting Limit.	Beginning and End of 12 hr. sequence or program specified.	70-130% or project specific/client limits	For results > 130% apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at the end and acceptable do not reject data.
Method Blank (MB)	1 per sample batch ≤ 20 samples of the same matrix	Analytes must not he higher than the highest of the following: 1/2 MRL, or 5% of the regulatory limit, or 5% of the associated sample concentration, or client specified conditions. QSM = ½ MRL	If sample is available and within holding times, sample associated with method blank needs to be reprepped. If no sample is available, qualify the data with a "B" to all associated positives when less than 5X blank concentration. QSM: Apply "B" to all results for the specific analytes in all samples in the associated preparatory batch.
Laboratory Control Sample (LCS)	1 per sample batch ≤ 20 samples of the same matrix	In house limits or client specified	If LCS fails percent recoveries. Re-analyze the LCS. If LCS recoveries are still outside QC control limits, if there is sample remaining, the sample batch must be reprepped. Qualify the failing analytes with a "Q". QSM: Correct problem, then repep and reanalyze the LCS and all samples in the associated batch for the failed analytes in the samples, if sufficient sample is available.
Matrix Spike (MS)	1 per sample batch ≤ 20 samples of the same matrix	In house limits or client specified	No action is taken based on MS recovery alone, use of professional judgement. For recoveries outside QC criteria, qualify out lying compound with "M". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.
Matrix Spike Duplicate (MSD)	1 per sample batch ≤ 20 samples of the same matrix	In house limits or client specified	No action is taken based on MSD results alone, use of professional judgement. If RPD is outside QC criteria, then qualify the out lying compounds with "Y". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.
Surrogates	Every sample and QC	In house limits or client specified	Rerun sample. If no apparent matrix interference noticed re-extract sample. If no sample is available, qualify the surrogate with "S". QSM: For QC and field samples; correct problem, reprep and re-analyze all failed samples or failed surrogates in the associated batch, if sufficient sample material is available. If no sample available, field samples apply "J" to the specific sample, QC samples apply "Q" to all associated samples.

Table 3
Summary of Method Objectives for Method 8082-PCBs by Aroclors

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Target Analyte Confirmation	Whenever a positive is detected, check agreement between primary and secondary columns.	RPD $\leq$ 40% QSM: Discuss in case narrative about qualified data.	**If not present**; QSM: Report positive value with a "Q" qualifier. Non-QSM: Do not report value. **If present and RPD $\leq$40%** QSM: Report higher of the two values. Non-QSM: Reporting the lower of the two values may be appropriate. **If present and RPD >40%** QSM: Report higher value with a "J" qualifier. Non-QSM: Report lower of the two values, may be appropriate, with a "P" qualifier.

Table 4

Analytical Run# **8082 PCB Analysis Data Review Checklist**

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the PCB initial calibration (Aroclor 1016/1260) performed using a minimum of five varying standard concentration levels on two dissimilar columns?	Lowest standard at or near MRL					
b. Is the variation between calibration response factors for all concentration levels <20% RSD, is r^2 >.990, or r > 0.995 for the regression line?	RSD <20%, r^2 >.990, r>0.995					
c. Was each ICAL uniquely identified (i.e. Standard Number)?						
d. Were there Calibration Factors (CF) established for the remaining Aroclors?						
d. Was an initial calibration blank (ICB) analyzed?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there second source ICVs for all Aroclors analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the recoveries for the ICVs within program limits?	%Recovery					
c. Was the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs (Aroclor 1016/1260) analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run? QSM = every ten sample injections.						
b. Were the recoveries for the CCVs within program limits?	%Recovery					
c. Were confirmed Aroclor detects processed using the appropriate Aroclor method?						
d. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8082 PCB Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was the method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch < 20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a . frequency one per Prep Batch?	Batch < 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	In-house limits or client specified limits					

Table 4
8082 PCB Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. acid treatment, florisil, and sulfur treatment)	Acid, Florisil, GPC, Sulfur Treatments					
f. Was there a hexane injection preformed prior to sample analysis?						
g. Was there a priming standard injected prior to sample analysis?						
9. RECORDS AND REPORTING						
a. Is the Analytical Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, analytical data, and method transfer to PDF format?						
b. Are all chromatograms stamped with initials and date?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier in LIMS and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Paragon Analytics
Method Implementation of EPA Method 6850
Perchlorate by HPLC/ESI//MS/MS

January 17, 2008

Introduction

Analytical determination of perchlorate ion concentration in soil/solid and water samples by HPLC/ESI/MS/MS was implemented at Paragon Analytics to provide high quality, sensitive and reliable analytical services to meet expressed client needs. The historical (ion chromatography) method, EPA 314, is not acceptable for many applications due to the relatively high occurrence of false positive results and significant quantitative interferences. The specificity of MS/MS effectively precludes the possibility of false positive results and associated high quantitative bias. The HPLC/ESI/MS/MS method also effectively precludes false negative results.

EPA method 6850 (multi-matrix) is a validated method. Development work was performed at Paragon Analytics to determine the exact instrument parameters and techniques to be used at Paragon Analytics to effectively implement method 6850 for subsequent routine analysis of water or solid matrix samples. A summary of parameters tested, observations and final parameters chosen for the standard operating procedure follows.

Method development and implementation were performed to ensure that analytical performance meets and exceeds the requirements of EPA Methods 6850, 331.0, DOD Perchlorate Handbook (Rev. 1 Change 1) and existing client quality goals expectations. A method detection limit (MDL) study and initial demonstration of capabilities (IDOC) (SOP 329 Rev. 6) were completed after the final method parameters were determined.

Sufficient sensitivity and instrument performance to meet the requirements of 331.0 were achieved. Development of the Paragon Analytics Program Specification and Nick Names will be necessary to complete implementation of method 331.0.

System Preparation

The HPLC/MS/MS system (Shimadzu Prominence/Applied Biosystems API 3200) was properly installed and documented to meet the manufacturer's specifications. Maintenance records were initiated to capture the installation and subsequent maintenance events. Paragon Analytics purchased training from the instrument manufacturer that was performed both at the manufacturer's facility and at the Paragon Analytics laboratory. The system was tuned (both mass calibration and analyte-specific parameters were optimized) prior to initiation of any analytical method (Appendix I Tune Package and Front End Optimization Data).

Evaluation of Alternate Mobile Phase Composition

Evaluation of higher aqueous content of mobile phase was performed to determine if improved control of RT could be accomplished. Mobile phase with the 80% H2O, 20%ACN and 0.1%HOAc was evaluated with anion challenge samples at 0, 500 and 2000ppm of anion-mix with 0.05, 1.0, and a 100ng/mL perchlorate. The 20% ACN yielded minimal RT shift with increasing anion concentration, however, increased peak splitting also accompanied higher anion and is unsuitable for a rugged assay. The 50% ACN MP was susceptible to greater RT shift with increasing anion, but showed less or no peak splitting or ghosting. The RT shift does not cause a problem for perchlorate identification due to equivalent elution of the $^{18}O_4$ perchlorate internal standard and adequate control of interferences was also accomplished using the 50% H2O, 50%ACN and 0.1%HOAc mobile phase (Appendix II).

Extract Composition and Peak Shape

Initial work included injection of aqueous extracts. Peak spitting was observed. The extracts were diluted to match or approximate the composition of the mobile phase. It was discovered that addition of 200 uL of ACN/HOAc (organic component of mobile phase) was sufficient to ensure good and consistent peak shape (Appendix III).

Column Temperature

Evaluation of column temperature was performed to determine the effect on separation of perchlorate from common interferences and to support the choice of a routine column temperature.

Evaluation of 20°C, 30°C, 40°C, 50°C column temperatures was performed to determine the optimal column operating temperature. Mobile phase composition was held constant at 50:50 Acetonitrile:Water with 0.1% acetic acid per SOP 448.

Table 1 Effect of Column Temperature and Anion Concentration on Perchlorate Retention Time (Appendix IV)

Temperature (°C)	Perchlorate Concentration (µg/L)	Anion Challenge Concentration (ppm)	Perchlorate Retention Time (RT in minutes)
20	1	0	15.99
20	1	500	14.36
20	1	2000	12.25
30	1	0	12.81
30	1	500	11.23
30	1	2000	9.48
40	1	0	11.13
40	1	500	9.62
40	1	2000	8.35
50	1	0	10.15
50	1	500	8.72
50	1	2000	7.42

Increasing the temperature of the column decreases the perchlorate retention time. The manufacturer's maximum recommended column temperature is 60 °C. Elevated temperatures caused decreased retention of perchlorate and did not provide method performance improvement. A nominal temperature setpoint of 30 °C was chosen to provide an acceptable balance between control of interference and total analytical run time.

Effect of Anion Challenge

A study to determine the maximum tolerable concentration of known ion suppressor ions bicarbonate, carbonate, chloride and sulfate was performed. This study included no divert event (see Table 1 results). Diversion of the HPLC effluent prior to elution of perchlorate may be used to protect the instrument from contamination. However, as sample conductivity increases, perchlorate retention decreases and perchlorate will go to divert at conductivity beyond about 17000 µS/cm. For this reason, 16620uS/cm (2000 mg/mL) is the level selected (Table 2) for the matrix conductivity threshold (MCT). Running an MCT sample containing a low concentration of perchlorate (as per SOP 448) can be performed to achieve a check of the instrument performance relative to matrix effects.

Table 2 Conductivity Limit Study Results

Conc. Suppressors (mg/mL)	Conductivity µS/cm	Suppression Observed	RT Drift
500	5000	No	Within Divert
2000	16620	No	Within Divert
4000	28500	No	Exceeds Divert
4500	31200	No	Exceeds Divert
5000	34000	IS < 50%	Exceeds Divert
5500	36700	IS < 50%	Exceeds Divert

Note: $^{35}ClO_3^-$ (m/z83)/ $^{35}ClO_3^-$(m/z85) ratio acceptable throughout study range.

Isotope ratios (m/z/ 83/85) were consistent throughout the study. Tabulated data acquired on 11/28/2007 and 12/11/2007 are included in Appendix IV. The range of ratio results across these data acquisitions, including all matrices and concentrations was 2.94 to 3.56 where our system produces a mean ratio of approximately 3.3. If a 20% window is applied to the 3.3 mean the resultant range is 2.64 to 3.96. The observed ratios are well within this range. Therefore, the observed performance will strongly support expected specificity of this LC/MS/MS method.

Sensitivity

The working quantitation range was determined as described in the linearity section below. Acceptable linearity (associated r value greater than 0.995) was demonstrated for the range of 0.01 to 100 µg/L. An MDL study was performed in accord with Paragon Analytics SOP (meets requirements of 40 CFR Part 136, Appendix B).

1. MDL
   A formal MDL study was conducted, submitted, reviewed and approved per Paragon Analytics procedural requirements. The complete study results are filed in Work Order Review/2007/MDL under M07074 (water) and M070075 (soil). A summary of the final results follows:

Table 3 Perchlorate MDL Results
Performed on 12/04/07 (expires 12/09/08); water and soil

Matrix	MDL (calculated)	MDL (to report)
Water	0.00397 µg/L	0.01 µg/L
Soil	0.0726 µg /kg	0.10 µg /kg

Note: A conservative MDL is to be reported to meet client requests for increased certainty in low-level results (per DOD Perchlorate Handbook Rev. 1 Change 1 and LANL/M. Minteer guidelines.)

2. **Reporting Limit:** The lowest concentration that can be routinely included in the multi-concentration calibration curve is typically used as the minimum RL. This limit must also be ≥ the DL. Although, the 0.01 µg/L standard is included in the calibration curve, the sensitivity limits (MDL and PRL) were elevated to limit reported low level quantitation error. If 0.01 µg/L is used as the MDL and 0.05 as the quantitation limit this goal is met. The accuracy of the 0.05 standard is significantly better than that of the 0.01 µg/L standard (approximately 30% error vs. 10 % error).

   Considerations to setting the MDL and reporting limit included:
   a. DOD Handbook: The concentration corresponding to the y-intercept value associated with the lowest calibration standard must be ≤ LOD. This criteria was observed to be met by the 0.01 µg/L standard.
   b. LANL: The signal to noise of the least abundant ion (m/z 85) should be greater than 25:1.
   c. LLOQ/LODV/DLV:
      i. 6850: RL standard ± 50%
      ii. DOD: Recover spike at 2x LOD ± 30%
      iii. LANL: Recover DL standard at ± 30% (and m/z 85 ≥ 25:1)

   The MDL limits as calculated and to be reported appear in Table 3. The limit of quantitation was set at 0.05 µg/L. The 0.05 µg/L standard was quantitated accurately against the curve. This interpretation meets both the SW846 and client expectations.

3. **Soil Detection Limits:** Soil detection limits were determined using a preparation that is identical to that used for field samples. The soil MDLs were chosen to be
   a. MDL: 0.1 µg/kg
   b. Minimum Reporting Limit: 0.5 µg/kg

Linearity

Solutions were prepared and analyzed at a broad array of concentrations to support evaluation of the linear response and working quantitation range of the system. From an interpretation of the observed responses, the optimal curve fit and concentrations to subsequently be used routinely were determined. A broad linear range was achieved. It was observed that use of additional concentration levels improved the curve fit and the quantitative accuracy. An example of concentrations associated with a successful calibration follows:

Example Calibration Curve:

Intermediate standards are prepared from 1000 µg/mL perchlorate stock (first source for calibration standards and second source for ICV) as follows:

Intermediate Perchlorate Solution A:

500 µg/L (50 µL of 1000µg/mL perchlorate to 100mL HPLC water)

Intermediate Perchlorate Solution B:

5 µg/L (100 µL of A to 10mL HPLC water)

Table 4 **CALIBRATION STANDARDS**

Level	Analyte Concentration (µg/L)	Standard Diluted (Intermediate Solutions Above)	Standard Added (µL)	Final Volume (mL)
ICAL 9	100	A: 500 µg/L Stock	2000	10.0
ICAL 8	50	A: 500 µg/L Stock	1000	10.0
ICAL 7	10	A: 500 µg/L Stock	200	10.0
ICAL 6	5.0	A: 500 µg/L Stock	100	10.0
ICAL 5	1.0	B: 5.0 µg/L	2000	10.0
ICAL 4	0.5	B: 5.0 µg/L	1000	10.0
ICAL 3	0.1	B: 5.0 µg/L	200	10.0
ICAL 2	0.05	B: 5.0 µg/L	100	10.0
ICAL 1	0.01	B: 5.0 µg/L	20	10.0
ICV	1.0	B: 5.0 µg/L (second source std.)	2000	10.0

Selectivity

Interferences and techniques to mitigate bias of perchlorate quantitative results were studied. Known potential interferences include bicarbonate, carbonate, chloride and sulfate. Any co-eluting compound that causes ionization suppression could limit method performance.

A study to determine the effect of anion challenge and to identify a daily challenge test was performed (Appendix IV). This is documented under *Effect of Anion Challenge* above. The study satisfied the intent of EPA Method 6850's MCT determination, of the interference threshold study (DOD Perchlorate Handbook) and of LANL's 500ppm ion challenge solution. The variation of the $^{35}ClO_3^-$ (m/z83)/$^{35}ClO_3^-$(m/z85) ratio is summarized above in the Effect of Anion Challenge section and tabulated data are included in Appendix IV. Anion ratios were stable and within the expected range. The results shown that Paragon Analytics' LC/ESI/MS/MS process provides excellent selectivity and that the effects of matrix challenge are well understood. Also, there exists sufficient sensitivity such that dilution may be adequate to resolve matrix challenges. If matrix challenges are present and dilution cannot correct the effect (i.e. low IS recovery) then additional cleanup procedures may be used. SPE C18 (for organic contamination) and/or cartridge cleanups such as Ba^{2+} (for sulfate), -H (for carbonate) and Ag (for chloride) may be used if removal of these interferences is appropriate. Two barium and one hydronium cartridge in series have been shown to be effective in removal of high sulfate and carbonate interferences. If cleanup columns are used, associated QC samples must be prepared in the same manner as the associated sample(s).

LIMS Reporting

Reporting of results using LIMs was established prior to release of the test sample package and prior to any routine sample analysis.

SOP

SOP was written and reviewed and submitted to Quality Assurance for publishing on January 14, 2008.

Performance Testing

Performance evaluation samples were analyzed and results found to be within acceptance limits. A client test package was also provided to demonstrate Paragon Analytics' Method 6850 capability. The performance testing results are posted in Workorder Review\QA\PT\2007\Stable\0711043 and included in Appendix IX.

IDOC

The IDOC requirements of Paragon Analytics SOP 329 Rev. 6 were completed for both water and soil matrices.

TYPICAL SYSTEM OPERATING CONDITIONS

HPLC Device	
Flow Rate (mobile phase):	0.5 mL/min
Auto Sampler Temperature	15°C
Injection Volume:	100 µL
Column Temperature:	30°C
Run Time:	17 min. (2 min. beyond peak tail)
Ionization Mode:	Turbo Spray (ESI)
Polarity:	Negative

Conclusion

The instrumentation was sourced and successfully installed within the timeline goal. Validation and implementation work demonstrated that the system performs within the expectations of EPA Method 6850 and that the additional criteria provided by the DOD Perchlorate Handbook and clients were also met. It is recommended that associated services be available to support client studies.

Appendices

I. Instrument Tune Package (mass calibration and perchlorate optimization) and Front End Optimization Data (11/27/07)
II. 80:20 (H2O: ACN) 0.1%HOAc Mobile Phase Evaluation and 50:50
III. Matching Mobile Phase Composition in Sample Extracts for Peak Share Optimization (ACN/HOAc addition to sample)
IV. Effect of Temperature and Anion Challenge Study
V. Effect of Anion Challenge on Baseline Noise
VI. Timeline of Development Activity
VII. Summary of EPA Method 6850 and Client Requirements
VIII. Performance Testing Result

References

EPA Method SW 6850 "Perchlorate in Waters, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS/MS)".

DOD Perchlorate Handbook; August 2007 Revision 1, Change 1

EPA 331.0 Rev. 1.0 January, 2005 "Determination of Perchlorate in Drinking water By Liquid Chromatography Electrospray Ionization Mass Spectrometry" EPA Document # 815-R-05-007.

SOP 329 Rev. 6 "Method Demonstration Procedures: Instrument Detection Limit (IDL)

Reviewed and Approved:

Linda Jefferies LC/MS/MS Specialist	1/22/08 Date
Steven Ignelzi Technical Specialist	1/21/2008 Date
Joel Nolte Organic Group Manager	1.21.08 Date
Deb Scheib Quality Control	3/20/08 Date

Appendix I

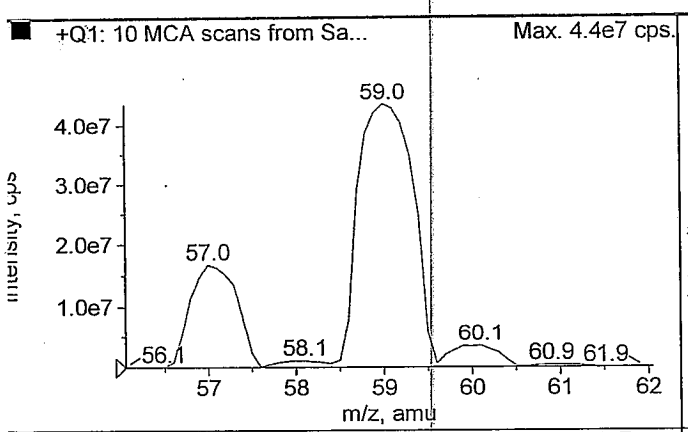
Instrument Tune Package
(mass calibration and perchlorate optimization)
and
Front End Optimization Data (11/27/07)

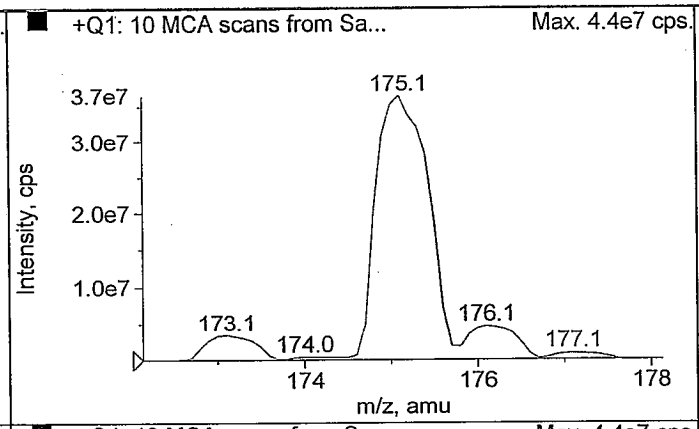
MASS CALIBRATION 10/09/07

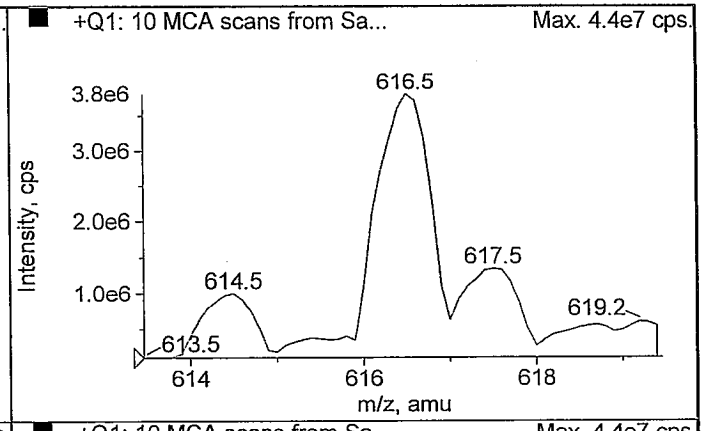
The following scans show mass calibration for Q1 and Q3 Positive PPG and Q1 and Q3 scans for Negative PPG.
Criteria:
Mass shift must be less than 0.1amu.
For unit resolution, a peak width of 0.7 ± 0.1 amu at 50% of the maximum peak height is acceptable.
Intensity (cps):

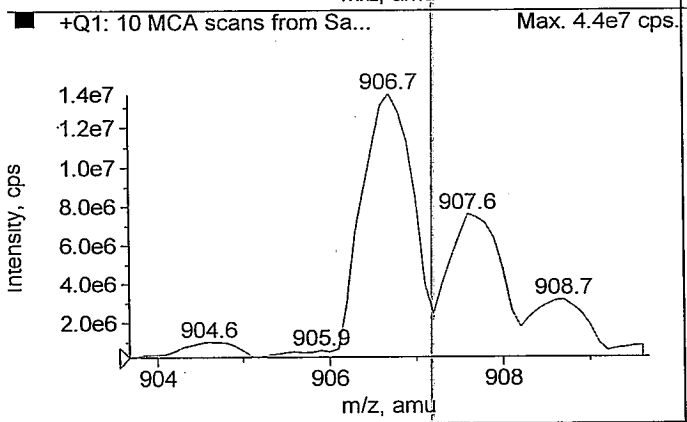
Q1 Positive, m/z 906.7$\geq 1.1 \times 10^7$cps, m/z 1545.1$\geq 2.7 \times 10^5$
Q3 Positive, m/z 906.7$\geq 8.1 \times 10^6$cps, m/z 1545.1$\geq 1.4 \times 10^5$

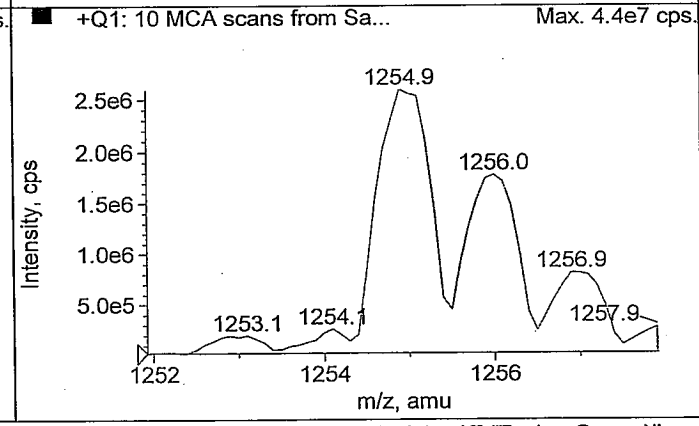
Q1 Negative, m/z 933.6$\geq 1.5 \times 10^6$cps, m/z 1572.1$\geq 1.4 \times 10^5$
Q3 Negative, m/z 933.6$\geq 7.2 \times 10^5$cps

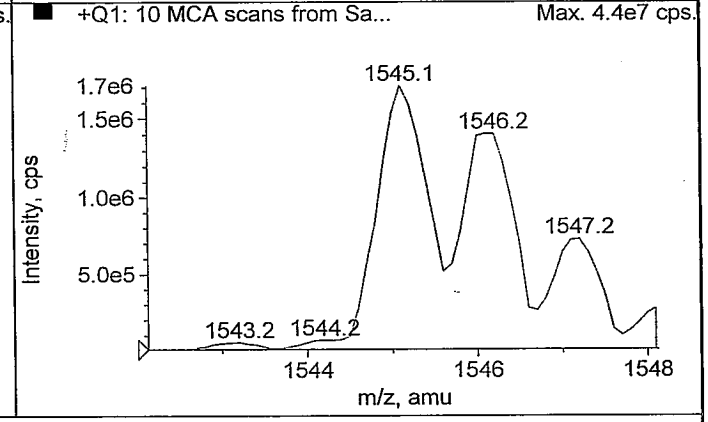






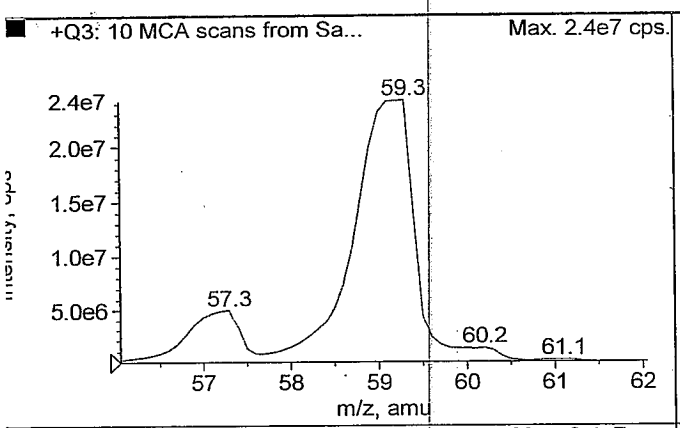


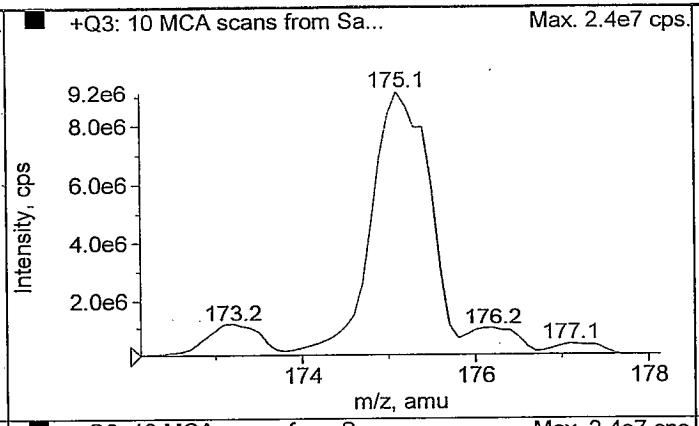


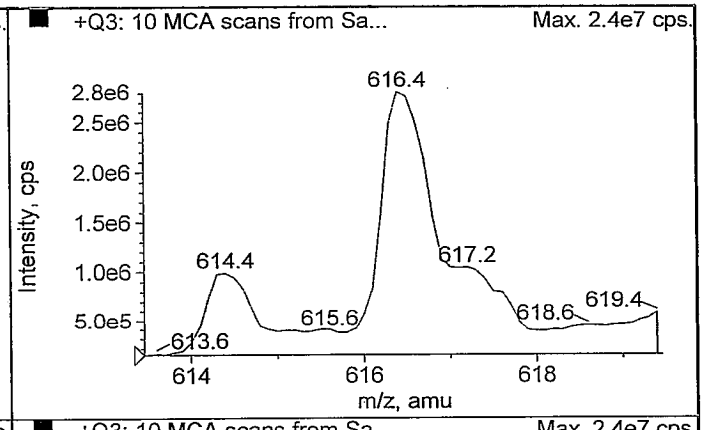


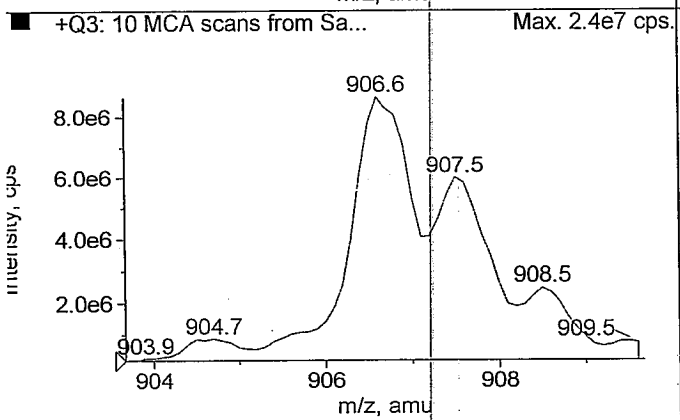
Peak List for " +Q1: 10 MCA scans from Sample 1 (TuneSampleID) of MT20071009121001.wiff (Turbo Spray)"

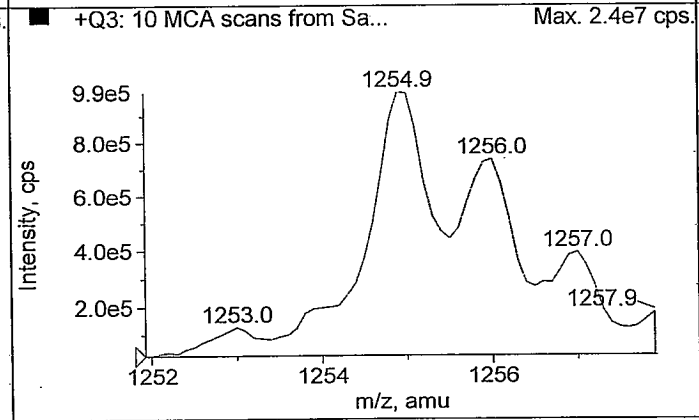
	Target Mass (amu)	Found At (amu)	Intensity (cps)	Width (amu)	Mass Shift (amu)
1	59.0500	59.0277	4.3567e7	0.7497	0.0223
2	175.1330	175.1307	3.6519e7	0.7225	2.3140e-3
3	616.4640	616.4790	3.8208e6	0.7525	-0.0150
4	906.6730	906.6953	1.3689e7	0.7267	-0.0223
5	1254.9250	1254.9524	2.5972e6	0.7506	-0.0274
6	1545.1340	1545.1318	1.7137e6	0.6804	2.2398e-3

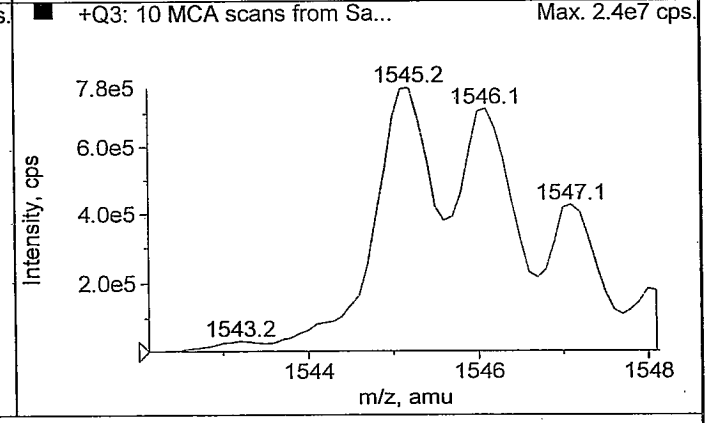






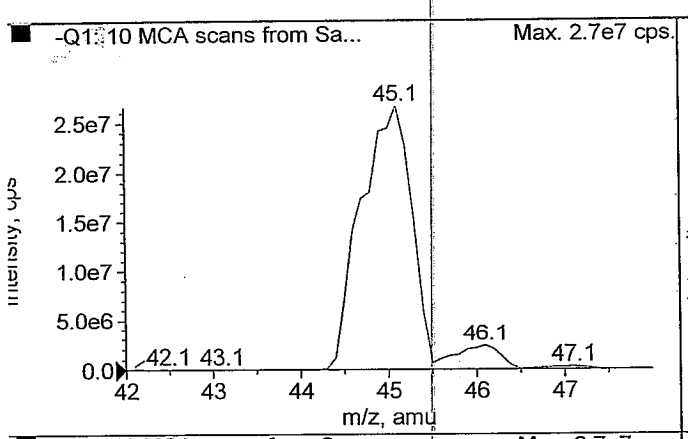


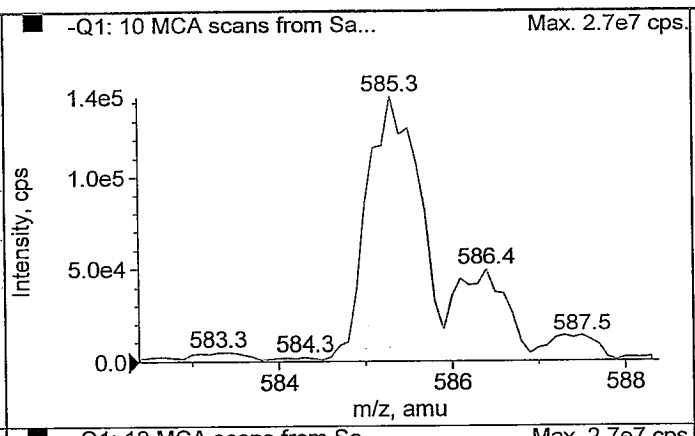


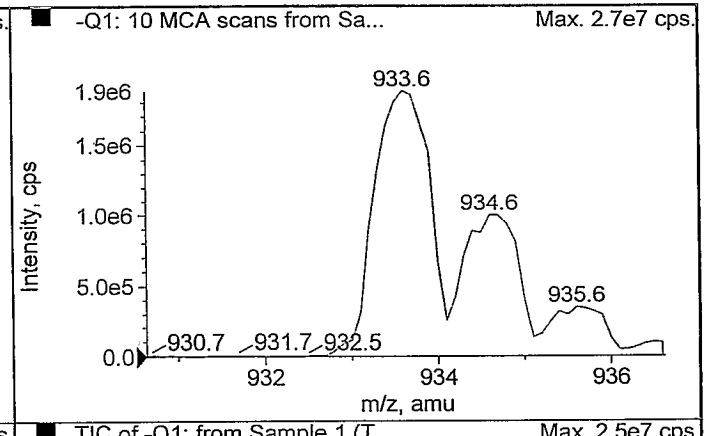


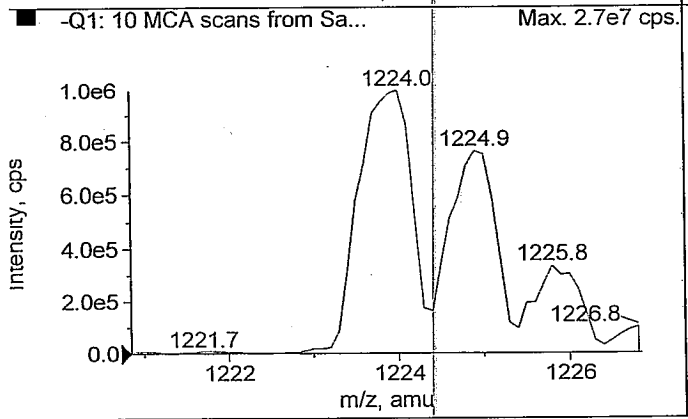
Peak List for " +Q3: 10 MCA scans from Sample 1 (TuneSampleID) of MT20071009101450.wiff (Turbo Spray)"

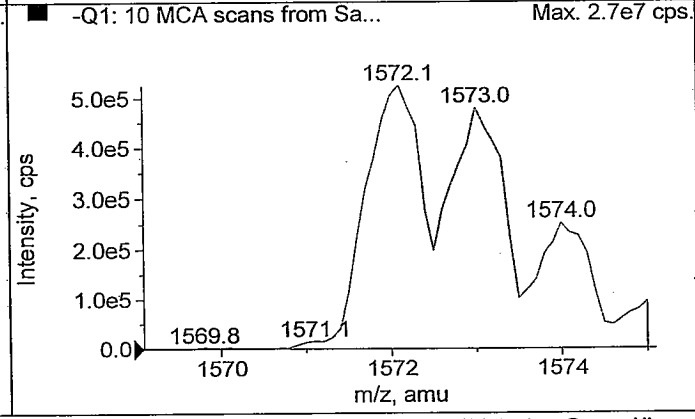
	Target Mass (amu)	Found At (amu)	Intensity (cps)	Width (amu)	Mass Shift (amu)
1	59.0500	59.1094	2.4309e7	0.6885	-0.0594
2	175.1330	175.1763	9.1530e6	0.7529	-0.0433
3	616.4640	616.4823	2.8113e6	0.6618	-0.0183
4	906.6730	906.6784	8.6559e6	0.7669	-5.3576e-3
5	1254.9250	1254.9476	9.8850e5	0.7647	-0.0226
6	1545.1340	1545.1607	7.7510e5	0.7825	-0.0267

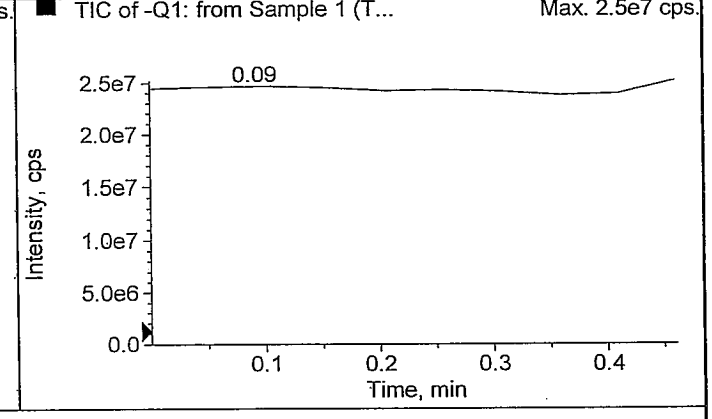






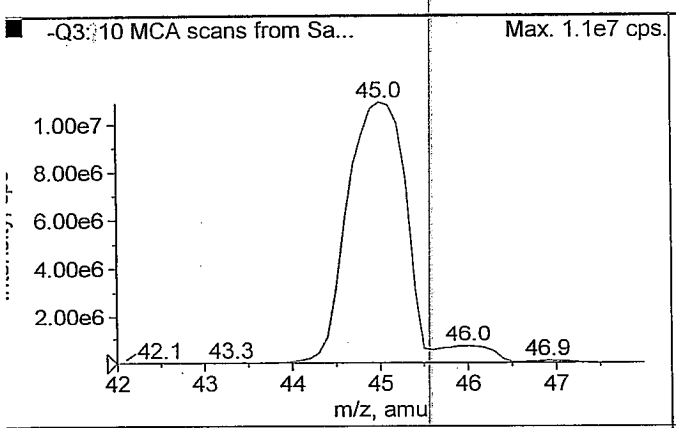


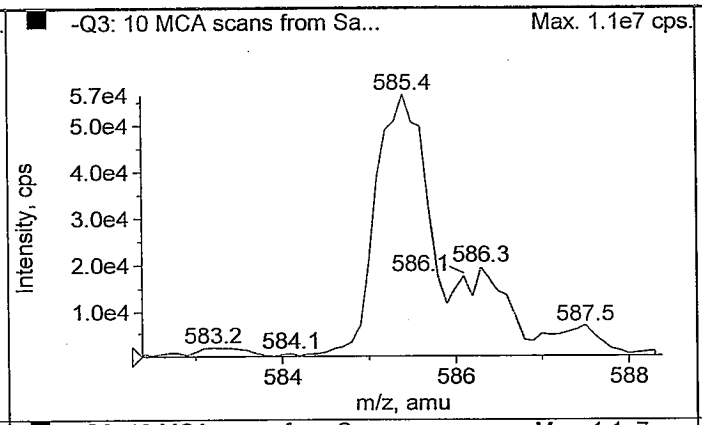


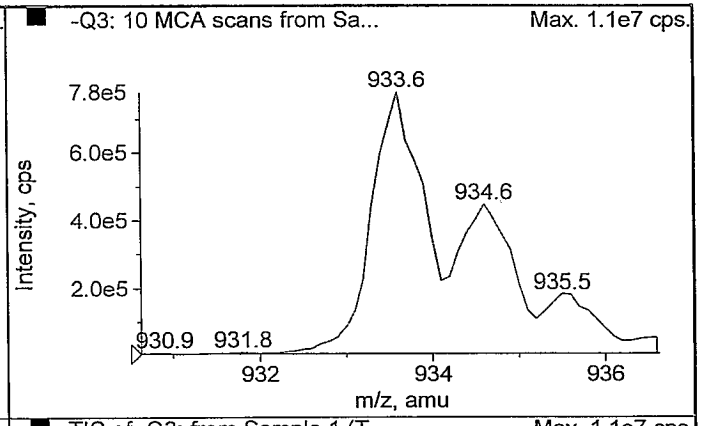


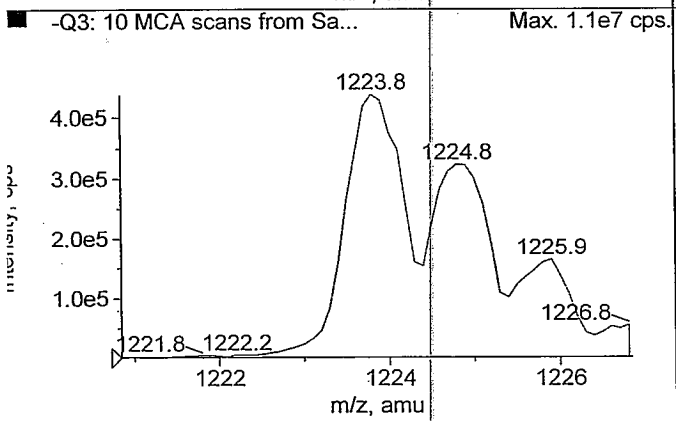
Peak List for " -Q1: 10 MCA scans from Sample 1 (TuneSampleID) of MT20071009103335.wiff (Turbo Spray)"

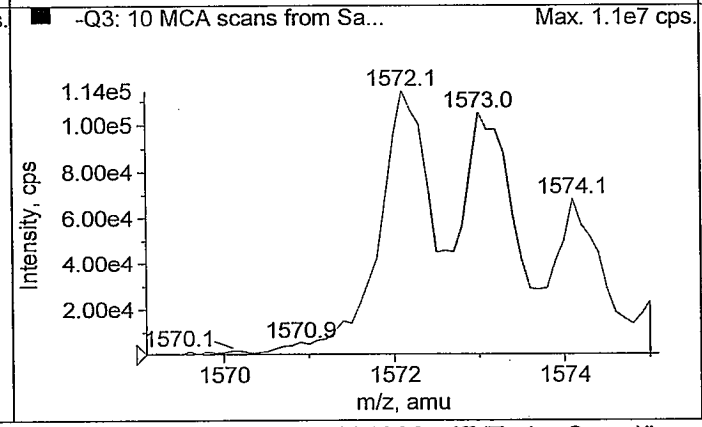
	Target Mass (amu)	Found At (amu)	Intensity (cps)	Width (amu)	Mass Shift (amu)
1	44.9980	45.0036	2.6624e7	0.7325	-5.6419e-3
2	585.3850	585.3385	1.4290e5	0.7489	0.0465
3	933.6360	933.6103	1.8773e6	0.7555	0.0257
4	1223.8450	1223.8615	9.9610e5	0.7350	-0.0165
5	1572.0970	1572.0560	5.2700e5	0.7770	0.0410

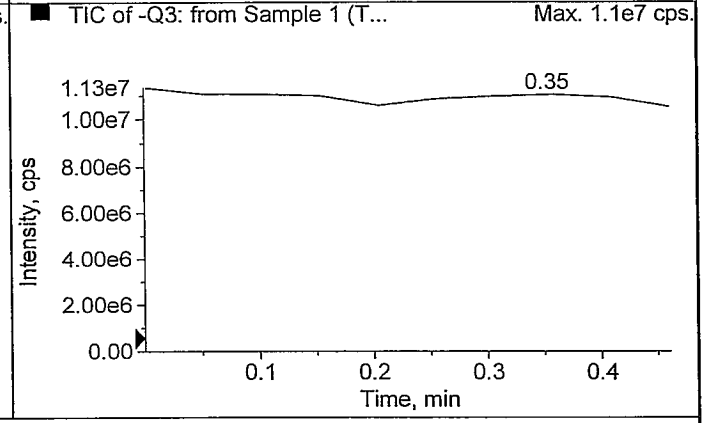












Peak List for " -Q3: 10 MCA scans from Sample 1 (TuneSampleID) of MT20071009104038.wiff (Turbo Spray)"

	Target Mass (amu)	Found At (amu)	Intensity (cps)	Width (amu)	Mass Shift (amu)
1	44.9980	44.9904	1.0904e7	0.7694	7.5670e-3
2	585.3850	585.3844	5.6600e4	0.6818	6.3521e-4
3	933.6360	933.6066	7.7990e5	0.6993	0.0294
4	1223.8450	1223.8407	4.3740e5	0.7823	4.3299e-3
5	1572.0970	1572.1571	1.1440e5	0.6029	-0.0601

PERCHLORATE TUNING from 12/18/2007

The following scans show Q1, Products of m/z 99 and 101 for Perchlorate and Q1 and Products of 107 scans for Perchlorate Internal Standard (IS).

Perchlorate Q1 shows precursor peaks of 99 and 101.
Products of 99 shows the expected product ion of 83 - from the 99 precursor.
Products of 101 shows the expected product ion of 85 - from the 101 precursor.

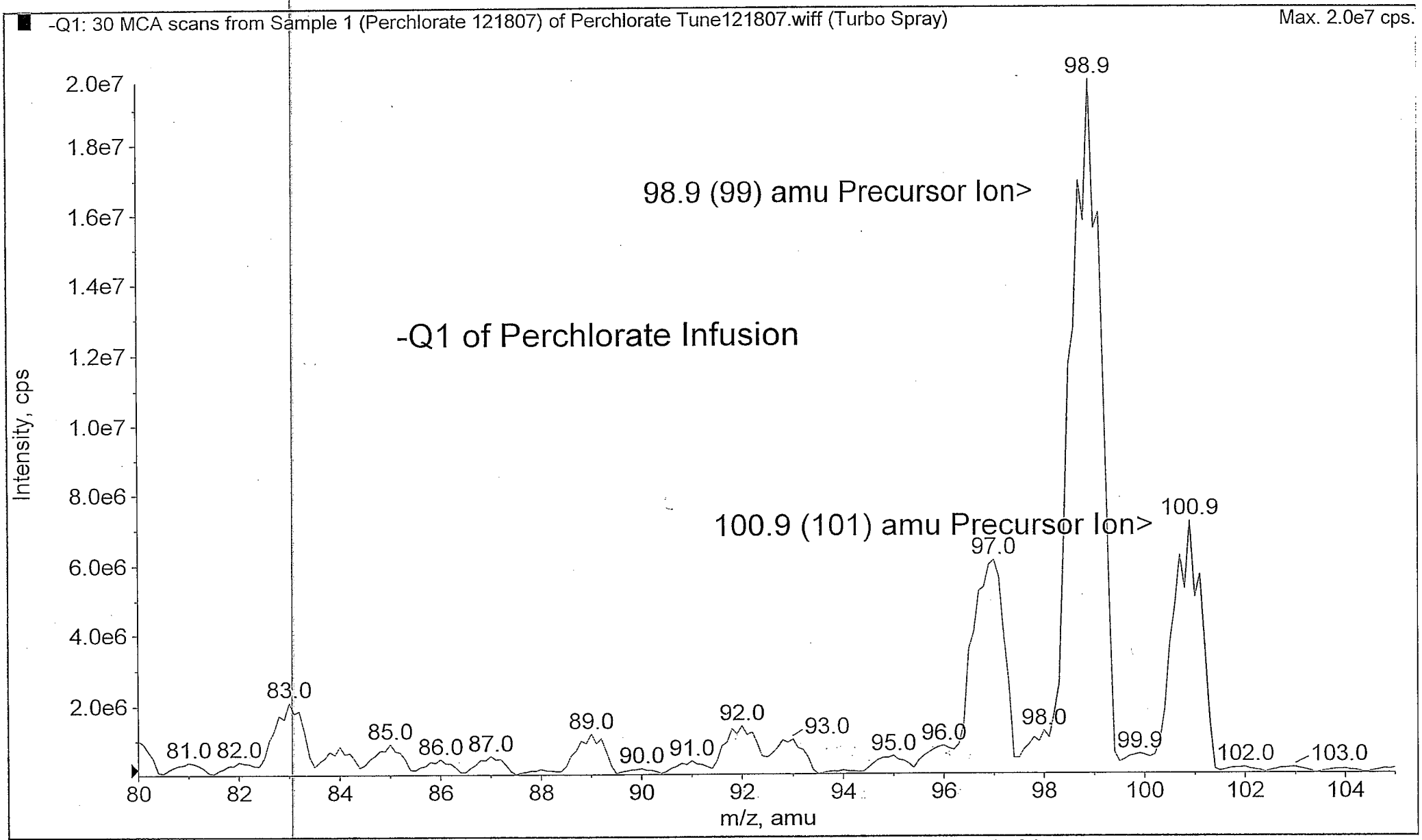
IS Perchlorate Q1 shows a precursor peak of 107.
IS Products of 107 shows the expected product ion of 89 from the 107 precursor.

In all cases, Precursors and Products are within $\pm$ 0.2 m/z of their expected masses.

Operator: jefferie
Printing Time: 2:26:32 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
Acq. Date: Tuesday, December 18, 2007
Acq. File: Perchlorate Tune121807.wiff

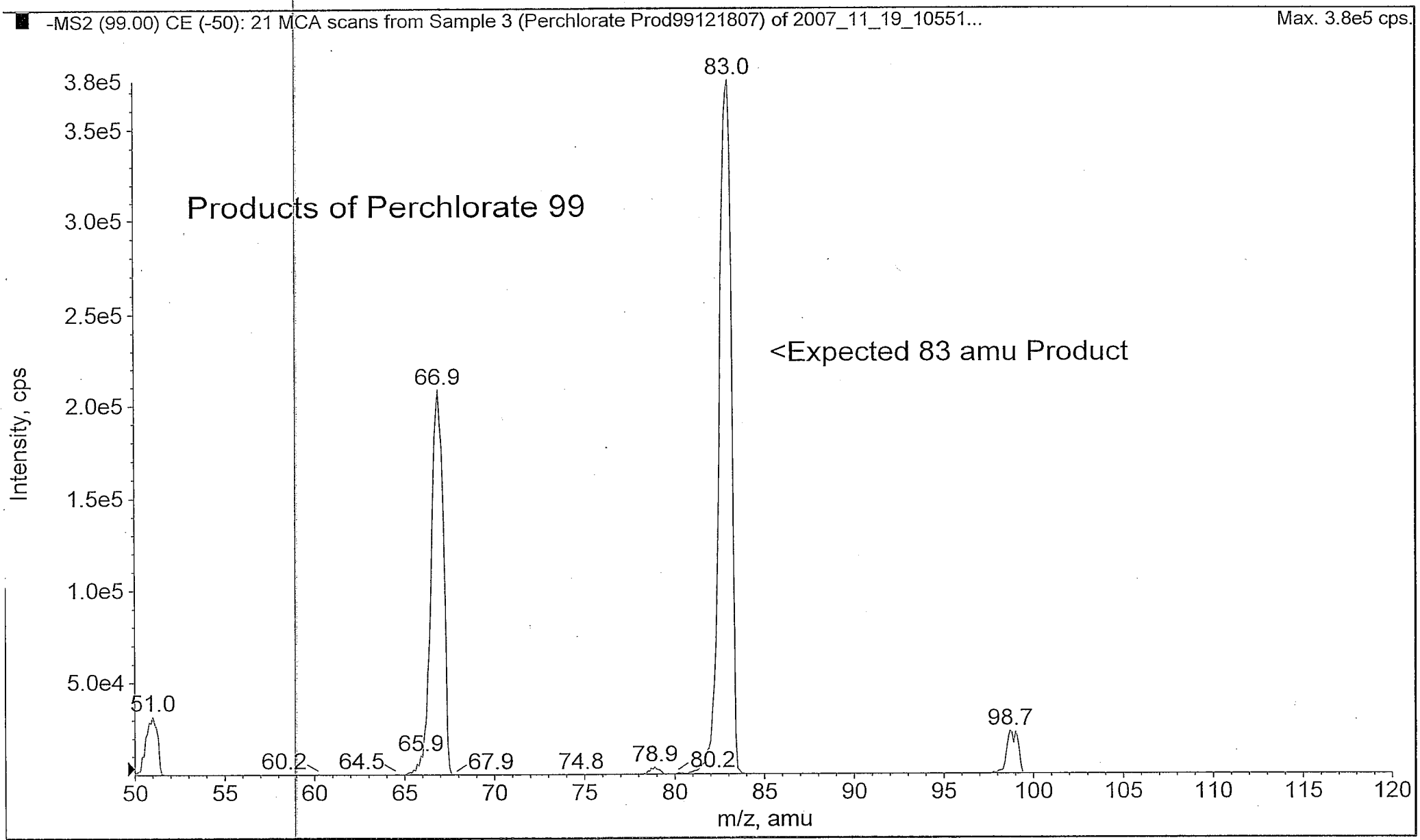


Workstation: LCMSMS

Operator: jefferie
Printing Time: 2:28:40 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
Acq. Date: Tuesday, December 18, 2007
Acq. File: 2007_11_19_105517_Perchlorate.wiff



Workstation: LCMSMS

Operator: jefferie
Printing Time: 2:35:08 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
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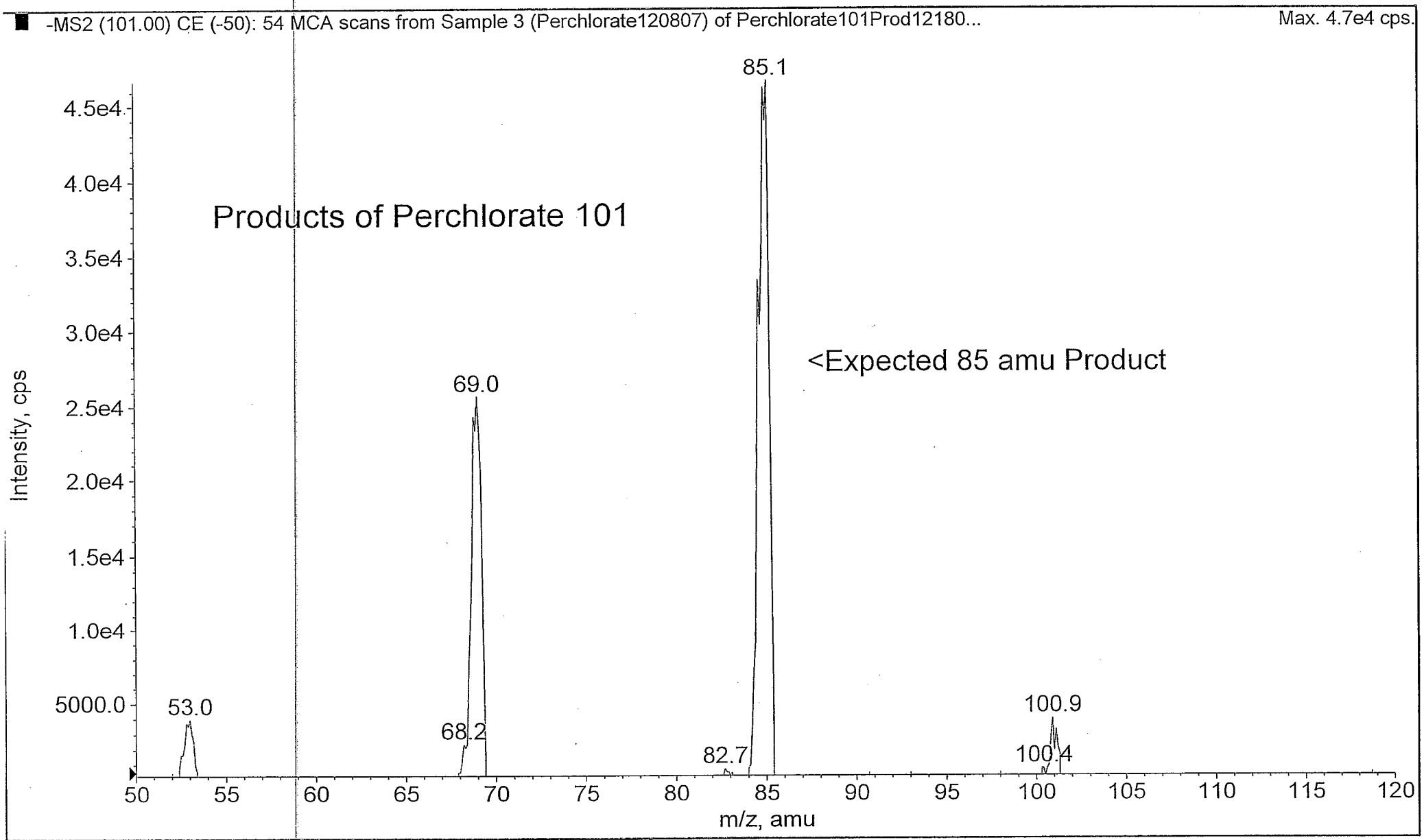
Acq. File: Perchlorate101Prod121807-2.wiff



Workstation: LCMSMS

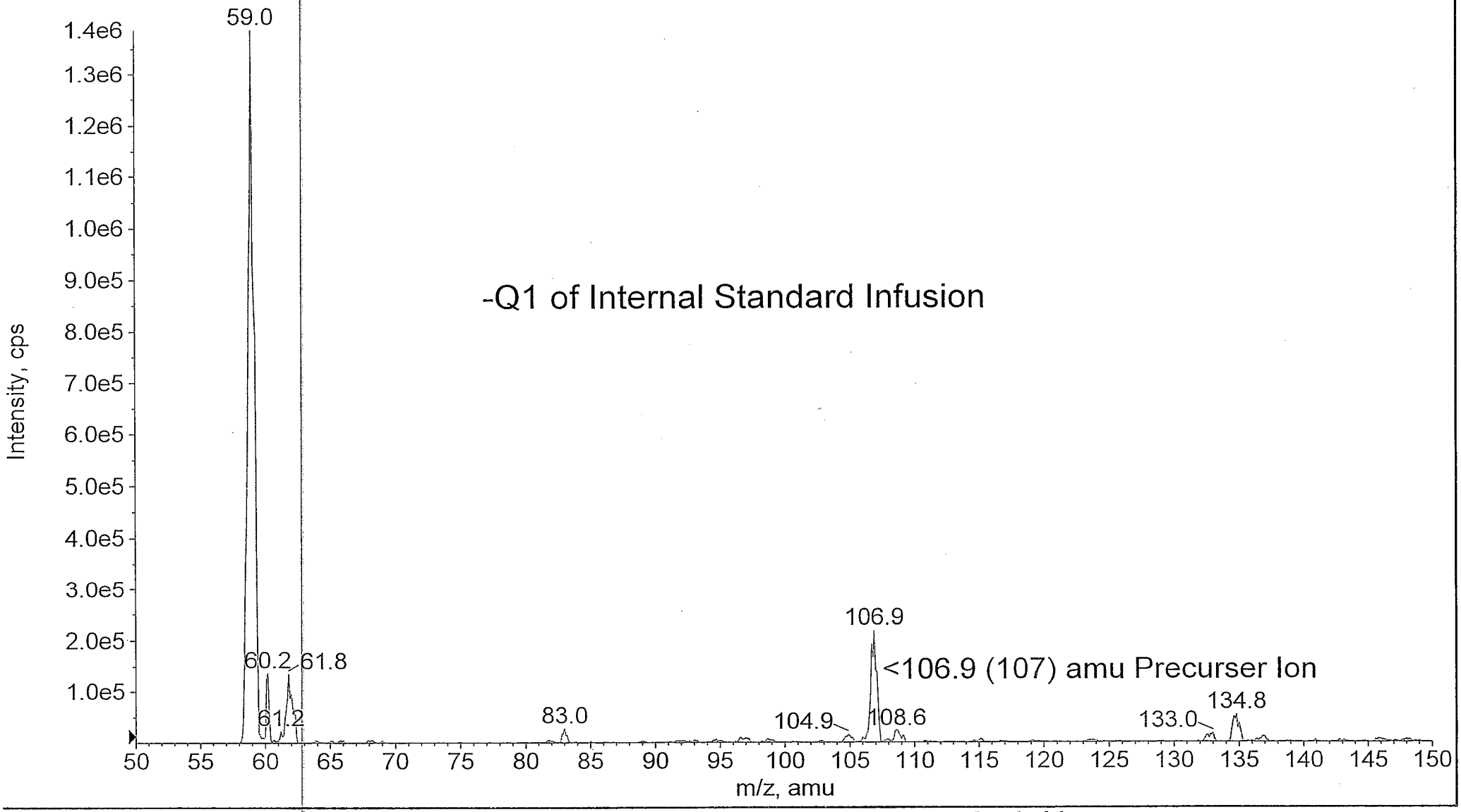
Operator: jefferie
Printing Time: 12:42:45 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
Acq. Date: Tuesday, December 18, 2007
Acq. File: PerchlorateIS121807.wiff

-Q1: 3.156 min from Sample 2 (ISQ1121807) of PerchlorateIS121807.wiff (Turbo Spray)

Max. 1.4e6 cps.

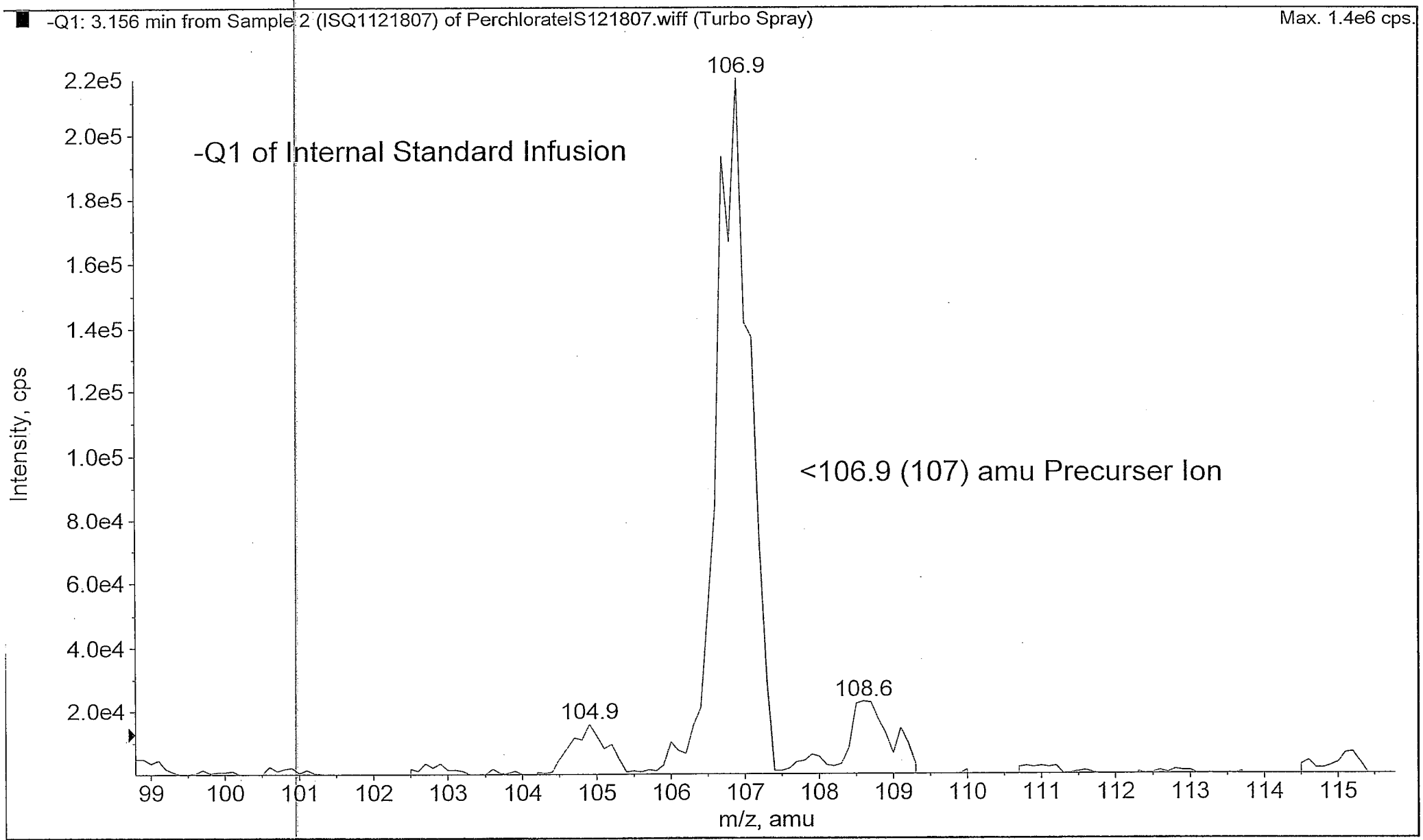


Workstation: LCMSMS

Operator: jefferie
Printing Time: 12:54:31 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
Acq. Date: Tuesday, December 18, 2007
Acq. File: PerchlorateIS121807.wiff

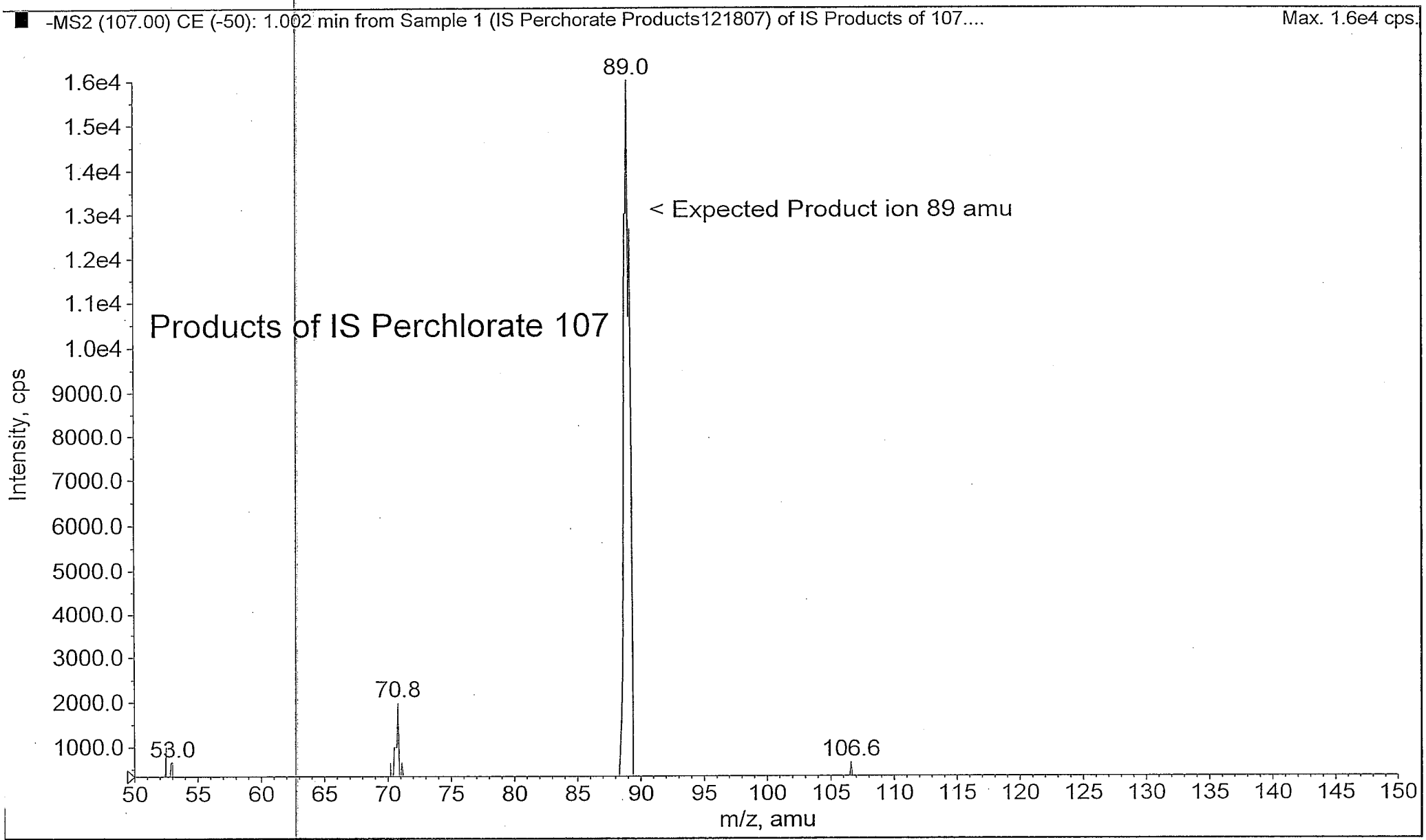


Workstation: LCMSMS

Operator: jefferie
Printing Time: 12:48:40 PM
Printing Date: Thursday, January 17, 2008

Project: Perchlorates
Algorithm: Analyst Classic

Batch Name: ManualTune.bat
Acq. Date: Tuesday, December 18, 2007
Acq. File: IS Products of 107.wiff



Workstation: LCMSMS

Front End Optimization: 11/27/07

Gas 1 - 55
Gas 2 - 75
CAD - 12
Curtain - 25
Temp - 575°C

Operator: jefferie
Printing Time: 12:08:37 PM
Printing Date: Tuesday, November 27, 2007

Shimadzu LC system Equlibration time = 0.00 min
Shimadzu LC system Injection Volume = 1.00 ul

State Parameter Editor

Ion Source:	Turbo Spray
Temperature Reached	
Curtain Gas (CUR):	10
Collision Gas (CAD):	5
IonSpray Voltage (IS):	-4500.0
Temperature (TEM):	575.0
Ion Source Gas 1 (GS1):	55
Ion Source Gas 2 (GS2):	75
Interface Heater (ihe):	On
Declustering Potential (DP):	-35.0
Entrance Potential (EP):	-10.0
Collision Energy (CE):	-34.0
Collision Cell Exit Potential (CXP):	0.0
Q1 Resolution:	UnitQ3 Resolution: Unit
Ion Energy 1 (IE1):	-0.8
Ion Energy 3 (IE3):	-1.5
Deflector (DF):	100.0

Shimadzu LC Method Parameters

Pumps

Pump A Model: LC-20AD
Pump B Model: LC-20AD
Pumping Mode: Isocratic Flow
Pump A Flow: 0.2500 mL/min.
Pump B Flow: 0.2500 mL/min.
Pressure Range (Pump A): 0 - 1422 psi
Pressure Range (Pump B): 0 - 1422 psi

Shimadzu LC Method Parameters

Autosampler

Model: SIL-20AC
Rinsing Volume: 200 uL
Needle Stroke: 52 mm.
Rinsing Speed: 35 uL/sec.
Sampling Speed: 15.0 uL/sec.
Purge Time: 25.0 min.
Rinse Dip Time: 0 sec.
Rinse Mode: No rinsing
Cooler Enabled: Yes
Cooler Temperature: 15 deg. C
Control Vial Needle Stroke: 52 mm

Oven

Model: CTO-20AC
Temperature Control: Disabled

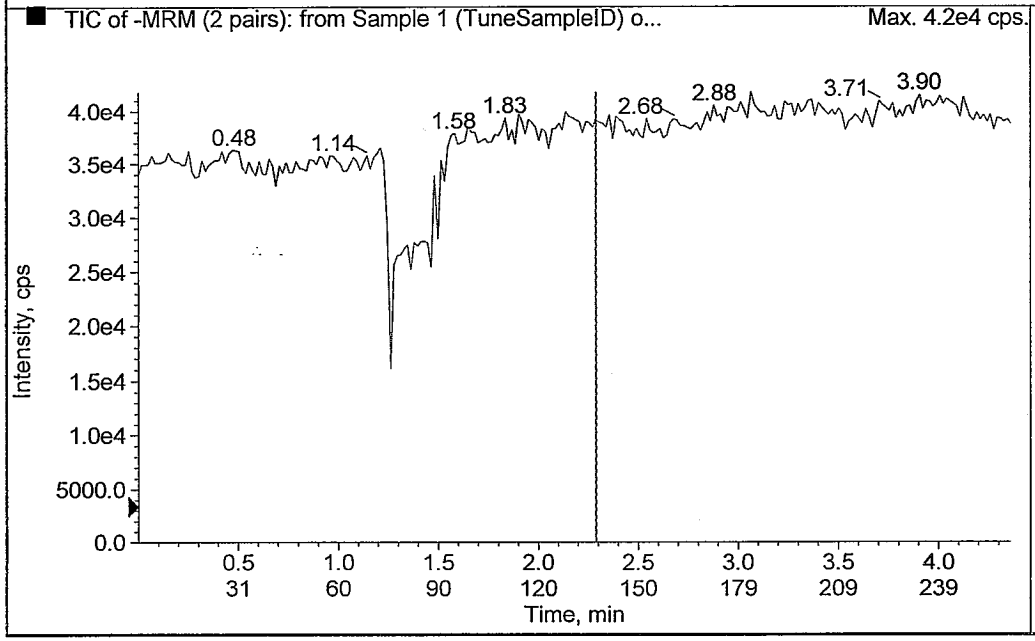
Shimadzu LC Method Parameters

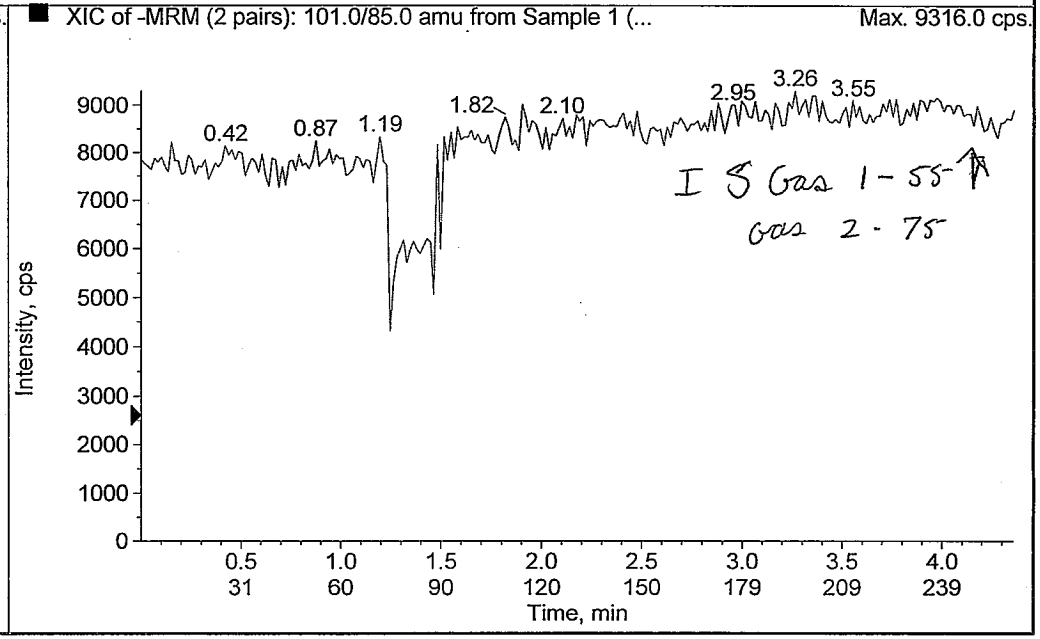
System Controller

Model: CBM-20A
Power: On
Event 1: Off
Event 2: Off
Event 3: Off
Event 4: Off

Time Program

Time	Module	Ev
5.01	System Controller	





Operator: jefferie
Printing Time: 11:58:36 AM
Printing Date: Tuesday, November 27, 2007

State Parameter Editor

Ion Source:	Turbo Spray
Temperature Reached	
Curtain Gas (CUR):	25.0
Collision Gas (CAD):	5
IonSpray Voltage (IS):	-4500.0
Temperature (TEM):	575.0
Ion Source Gas 1 (GS1):	60
Ion Source Gas 2 (GS2):	40
Interface Heater (ihe):	On
Declustering Potential (DP):	-35.0
Entrance Potential (EP):	-10.0
Collision Energy (CE):	-34.0
Collision Cell Exit Potential (CXP):	0.0
Q1 Resolution:	Unit
Q3 Resolution:	Unit
Ion Energy 1 (IE1):	-0.8
Ion Energy 3 (IE3):	-1.5
Deflector (DF):	100.0

Shimadzu LC system Equlibration time = 0.00 min
Shimadzu LC system Injection Volume = 1.00 ul
Shimadzu LC Method Parameters

Pumps

Pump A Model: LC-20AD
Pump B Model: LC-20AD
Pumping Mode: Isocratic Flow
Pump A Flow: 0.2500 mL/min.
Pump B Flow: 0.2500 mL/min.
Pressure Range (Pump A): 0 - 1422 psi
Pressure Range (Pump B): 0 - 1422 psi

Shimadzu LC Method Parameters

Autosampler

Model: SIL-20AC
Rinsing Volume: 200 uL
Needle Stroke: 52 mm.
Rinsing Speed: 35 uL/sec.
Sampling Speed: 15.0 uL/sec.
Purge Time: 25.0 min.
Rinse Dip Time: 0 sec.
Rinse Mode: No rinsing
Cooler Enabled: Yes
Cooler Temperature: 15 deg. C
Control Vial Needle Stroke: 52 mm

Oven

Model: CTO-20AC
Temperature Control: Disabled

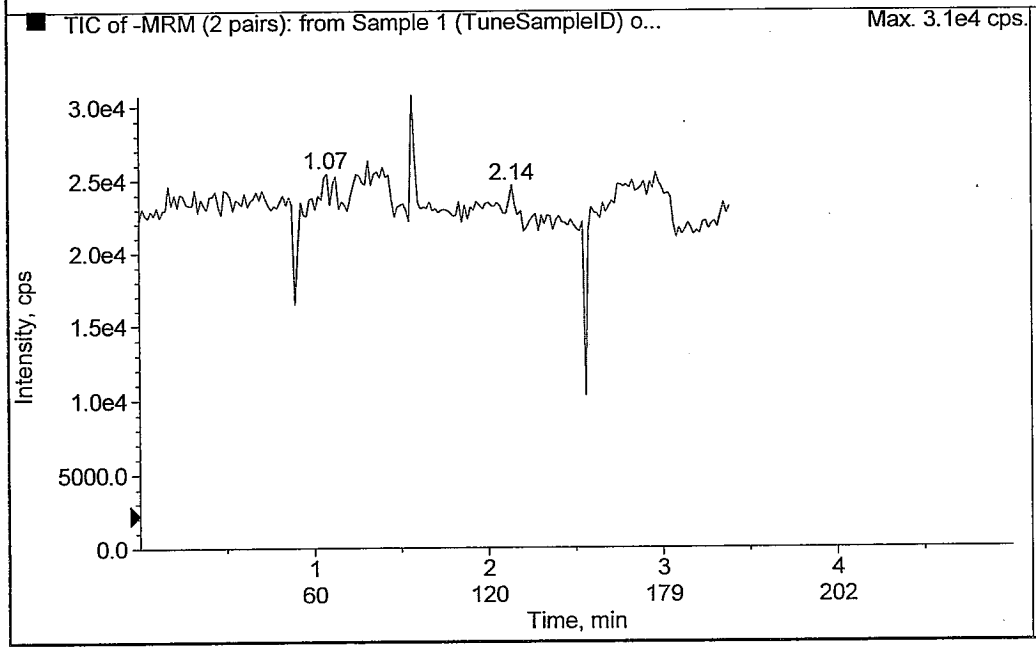
Shimadzu LC Method Parameters

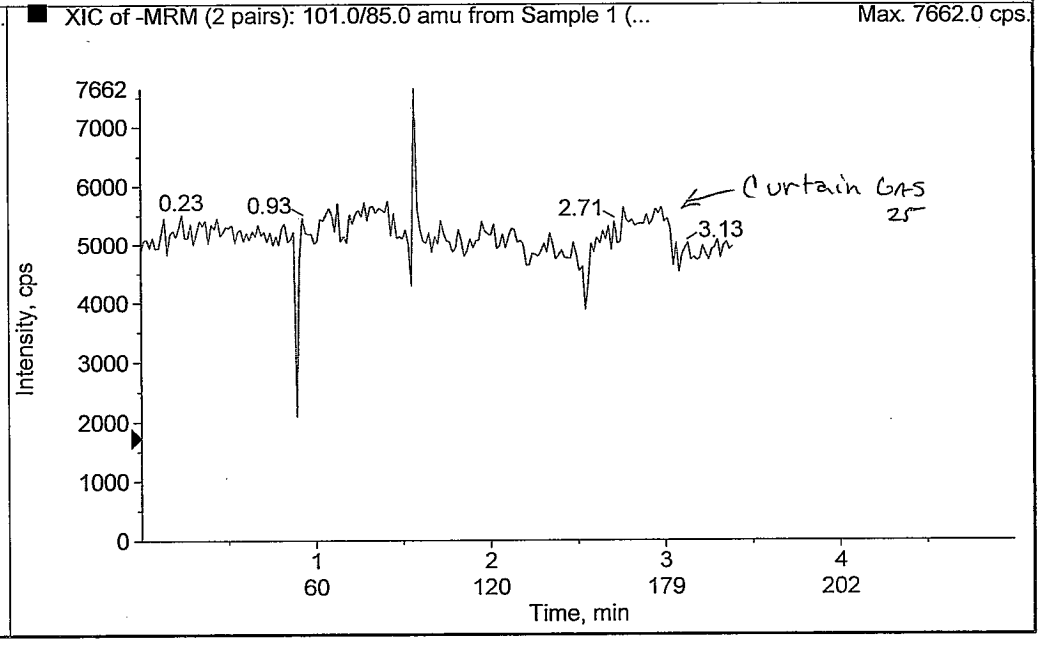
System Controller

Model: CBM-20A
Power: On
Event 1: Off
Event 2: Off
Event 3: Off
Event 4: Off

Time Program

Time	Module	Ev
5.01	System Controller	





Operator: jefferie
Printing Time: 12:26:11 PM
Printing Date: Tuesday, November 27, 2007

Shimadzu LC system Equlibration time = 0.00 min
Shimadzu LC system Injection Volume = 1.00 ul

Sate Parameter Editor

Ion Source:	Turbo Spray
Temperature Reached	
Curtain Gas (CUR):	25.0
Collision Gas (CAD):	12
IonSpray Voltage (IS):	-4500.0
Temperature (TEM):	600
Ion Source Gas 1 (GS1):	55.0
Ion Source Gas 2 (GS2):	75.0
Interface Heater (ihe):	On
Declustering Potential (DP):	-35.0
Entrance Potential (EP):	-10.0
Collision Energy (CE):	-34.0
Collision Cell Exit Potential (CXP):	0.0
Q1 Resolution:	Unit
Q3 Resolution:	Unit
Ion Energy 1 (IE1):	-0.8
Ion Energy 3 (IE3):	-1.5
Deflector (DF):	100.0

Shimadzu LC Method Parameters

Pumps

Pump A Model: LC-20AD
Pump B Model: LC-20AD
Pumping Mode: Isocratic Flow
Pump A Flow: 0.2500 mL/min.
Pump B Flow: 0.2500 mL/min.
Pressure Range (Pump A): 0 - 1422 psi
Pressure Range (Pump B): 0 - 1422 psi

Shimadzu LC Method Parameters

Autosampler

Model: SIL-20AC
Rinsing Volume: 200 uL
Needle Stroke: 52 mm.
Rinsing Speed: 35 uL/sec.
Sampling Speed: 15.0 uL/sec.
Purge Time: 25.0 min.
Rinse Dip Time: 0 sec.
Rinse Mode: No rinsing
Cooler Enabled: Yes
Cooler Temperature: 15 deg. C
Control Vial Needle Stroke: 52 mm

Oven

Model: CTO-20AC
Temperature Control: Disabled

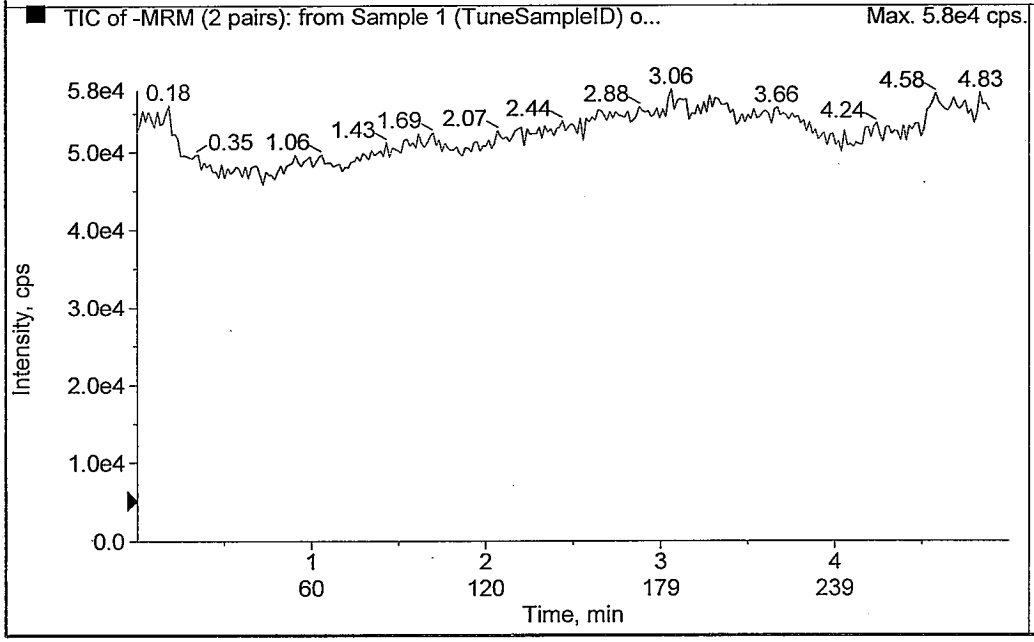
Shimadzu LC Method Parameters

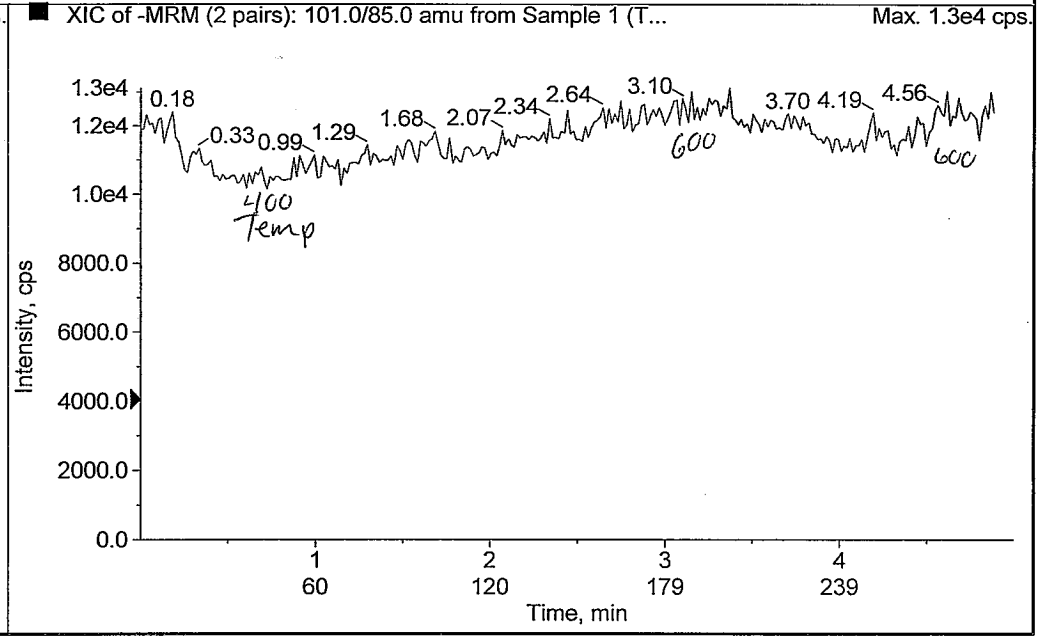
System Controller

Model: CBM-20A
Power: On
Event 1: Off
Event 2: Off
Event 3: Off
Event 4: Off

Time Program

Time	Module	Ev
5.01	System Controller	





Operator: jefferie
Printing Time: 12:14:44 PM
Printing Date: Tuesday, November 27, 2007

State Parameter Editor

Ion Source:	Turbo Spray
Temperature Reached	
Curtain Gas (CUR):	25.0
Collision Gas (CAD):	12
IonSpray Voltage (IS):	-4500.0
Temperature (TEM):	575.0
Ion Source Gas 1 (GS1):	55.0
Ion Source Gas 2 (GS2):	75.0
Interface Heater (ihe):	On
Declustering Potential (DP):	-35.0
Entrance Potential (EP):	-10.0
Collision Energy (CE):	-34.0
Collision Cell Exit Potential (CXP):	0.0
Q1 Resolution:	Unit
Q3 Resolution:	Unit
Ion Energy 1 (IE1):	-0.8
Ion Energy 3 (IE3):	-1.5
Deflector (DF):	100.0

Shimadzu LC system Equlibration time = 0.00 min
Shimadzu LC system Injection Volume = 1.00 ul
Shimadzu LC Method Parameters

Pumps

Pump A Model: LC-20AD
Pump B Model: LC-20AD
Pumping Mode: Isocratic Flow
Pump A Flow: 0.2500 mL/min.
Pump B Flow: 0.2500 mL/min.
Pressure Range (Pump A): 0 - 1422 psi
Pressure Range (Pump B): 0 - 1422 psi

Shimadzu LC Method Parameters

Autosampler

Model: SIL-20AC
Rinsing Volume: 200 uL
Needle Stroke: 52 mm.
Rinsing Speed: 35 uL/sec.
Sampling Speed: 15.0 uL/sec.
Purge Time: 25.0 min.
Rinse Dip Time: 0 sec.
Rinse Mode: No rinsing
Cooler Enabled: Yes
Cooler Temperature: 15 deg. C
Control Vial Needle Stroke: 52 mm

Oven

Model: CTO-20AC
Temperature Control: Disabled

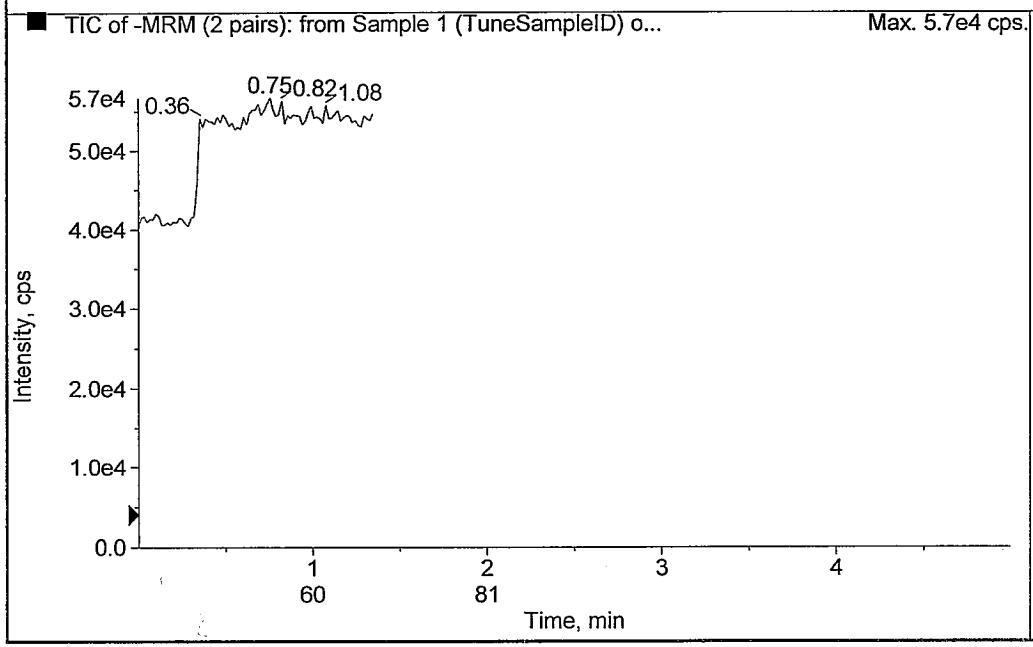
Shimadzu LC Method Parameters

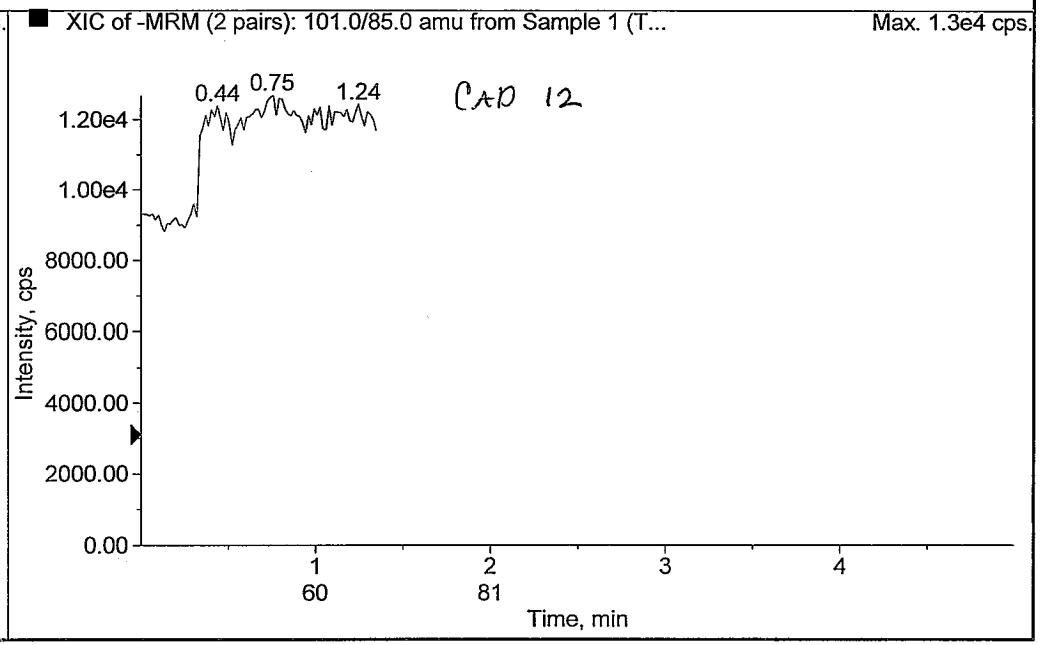
System Controller

Model: CBM-20A
Power: On
Event 1: Off
Event 2: Off
Event 3: Off
Event 4: Off

Time Program

Time	Module	Ev
5.01	System Controller	





Appendix II

Mobile Phase Evaluation

80:20 (H2O: ACN) 0.1%HOAc

50:50 (H2O: ACN) 0.1%HOAc

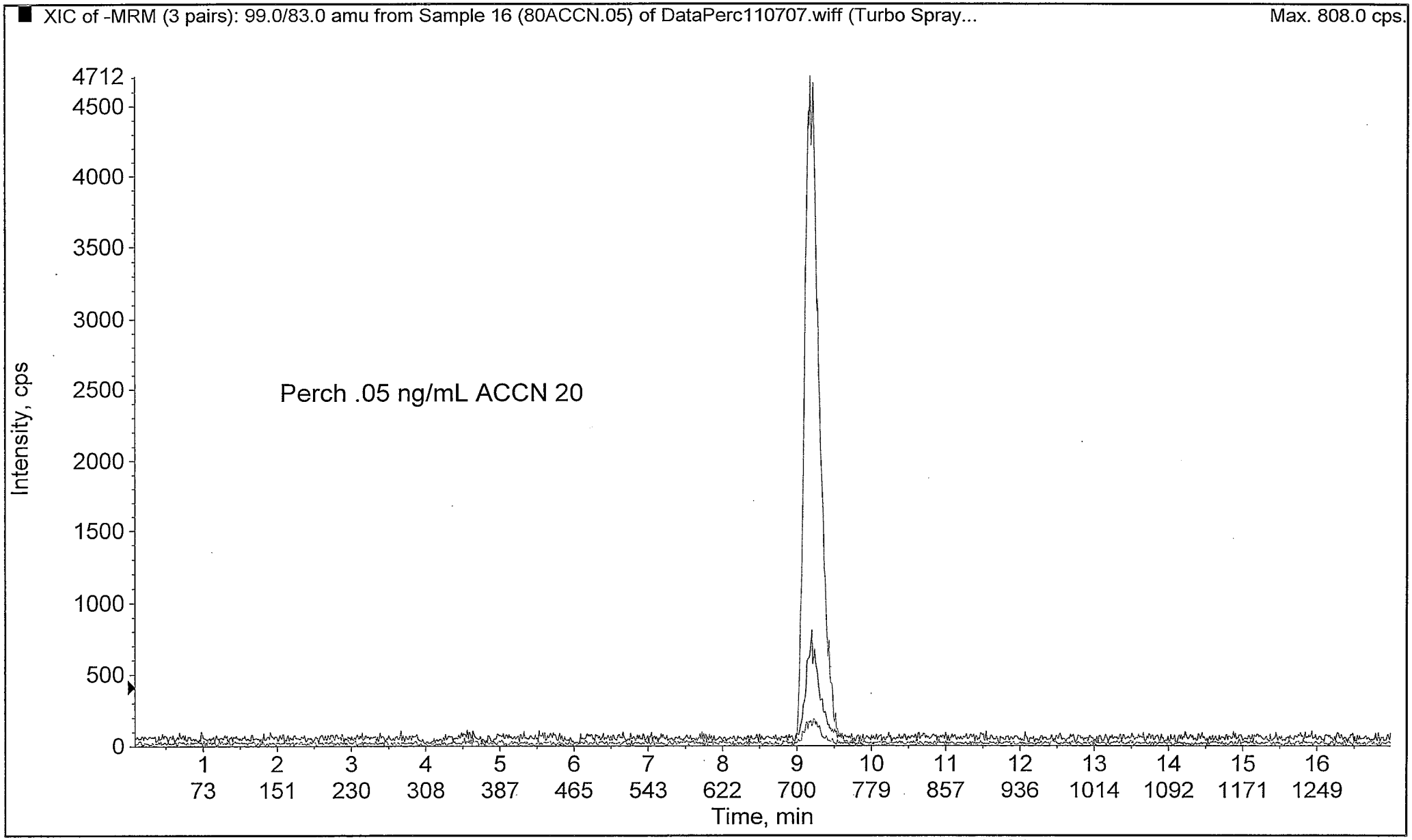
Operator: jefferie
Printing Time: 2:14:02 PM

Printing Date: Monday, January 21, 2008

Project: Perchlorates
Algorithm: Analyst Classic

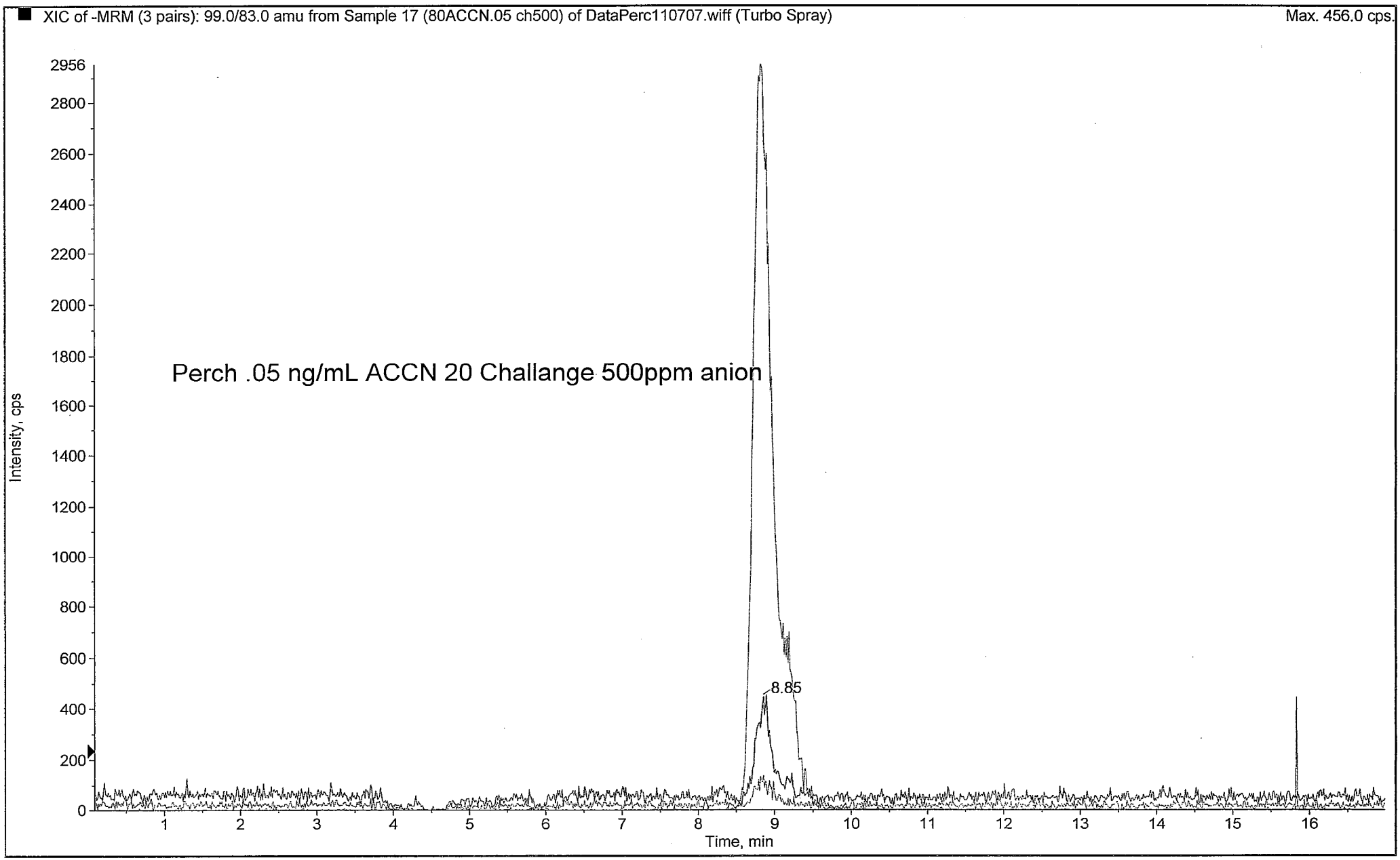
Batch Name: Perc110707.dab
Acq. Date: Thursday, November 08, 2007

Acq. File: DataPerc110707.wiff

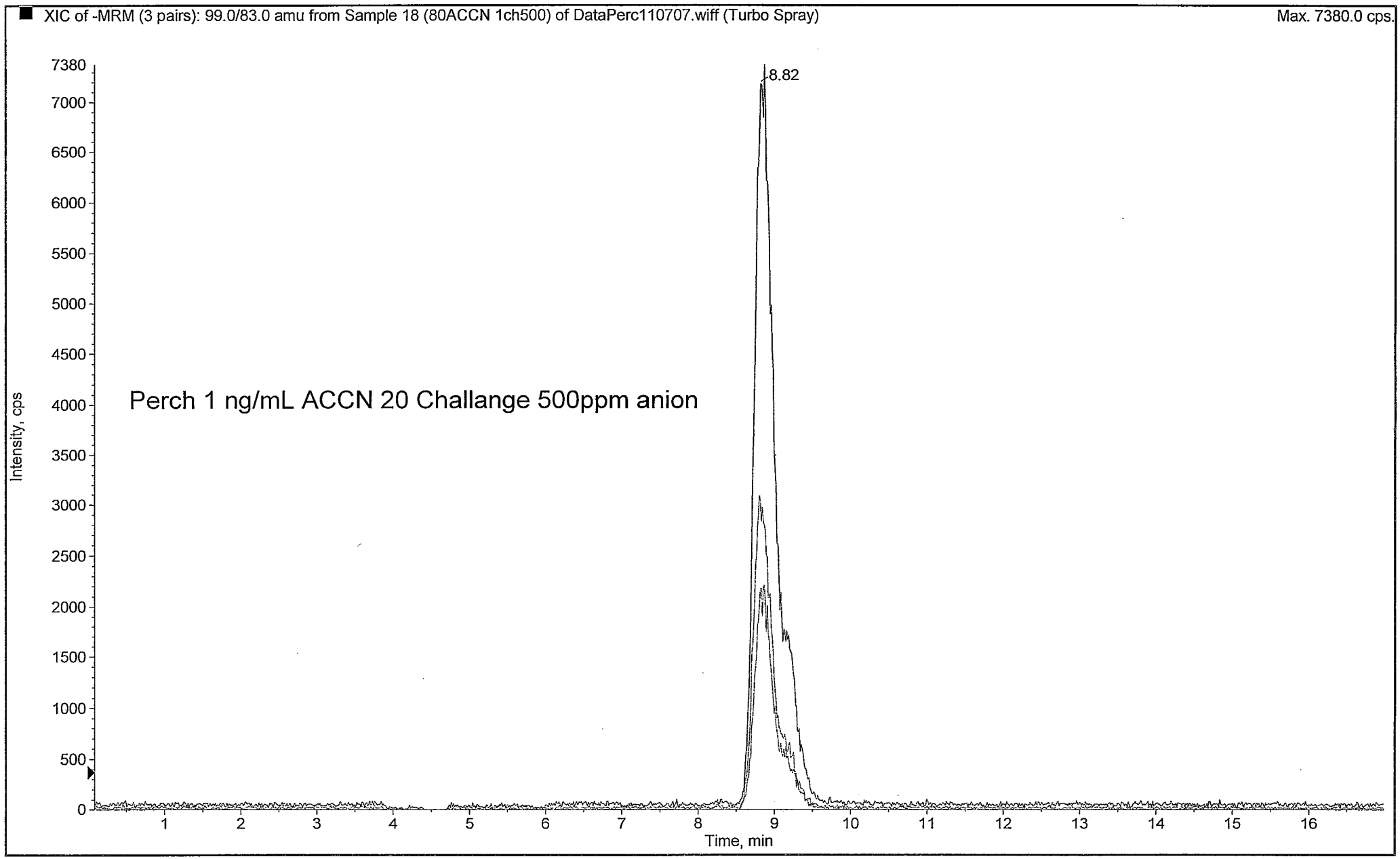


Workstation: LCMSMS

Operator: jefferie
Printing Time: 12:11:19 PM
Printing Date: Thursday, November 08, 2007



Operator: jefferie
Printing Time: 12:11:43 PM
Printing Date: Thursday, November 08, 2007



Operator: jefferie
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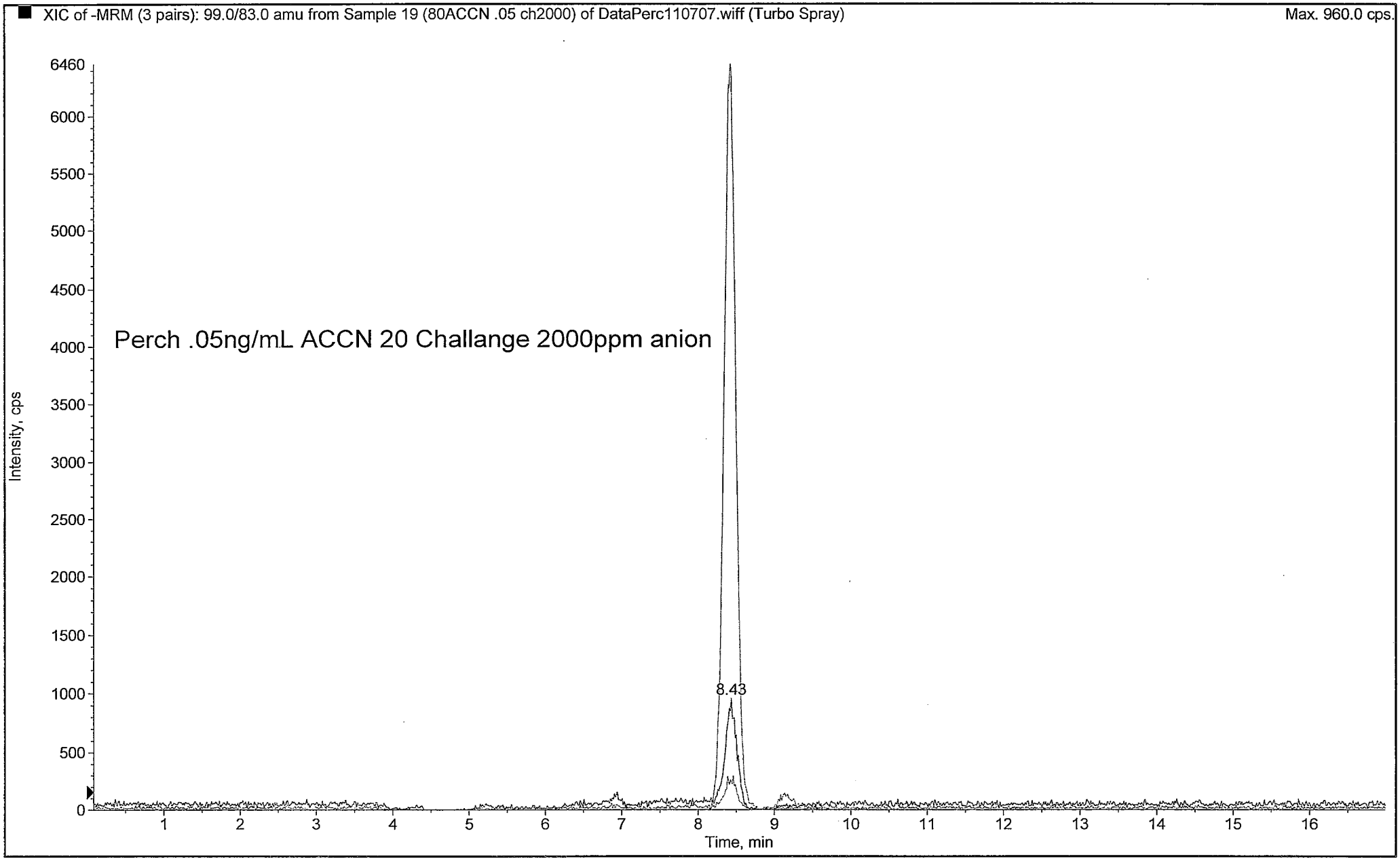
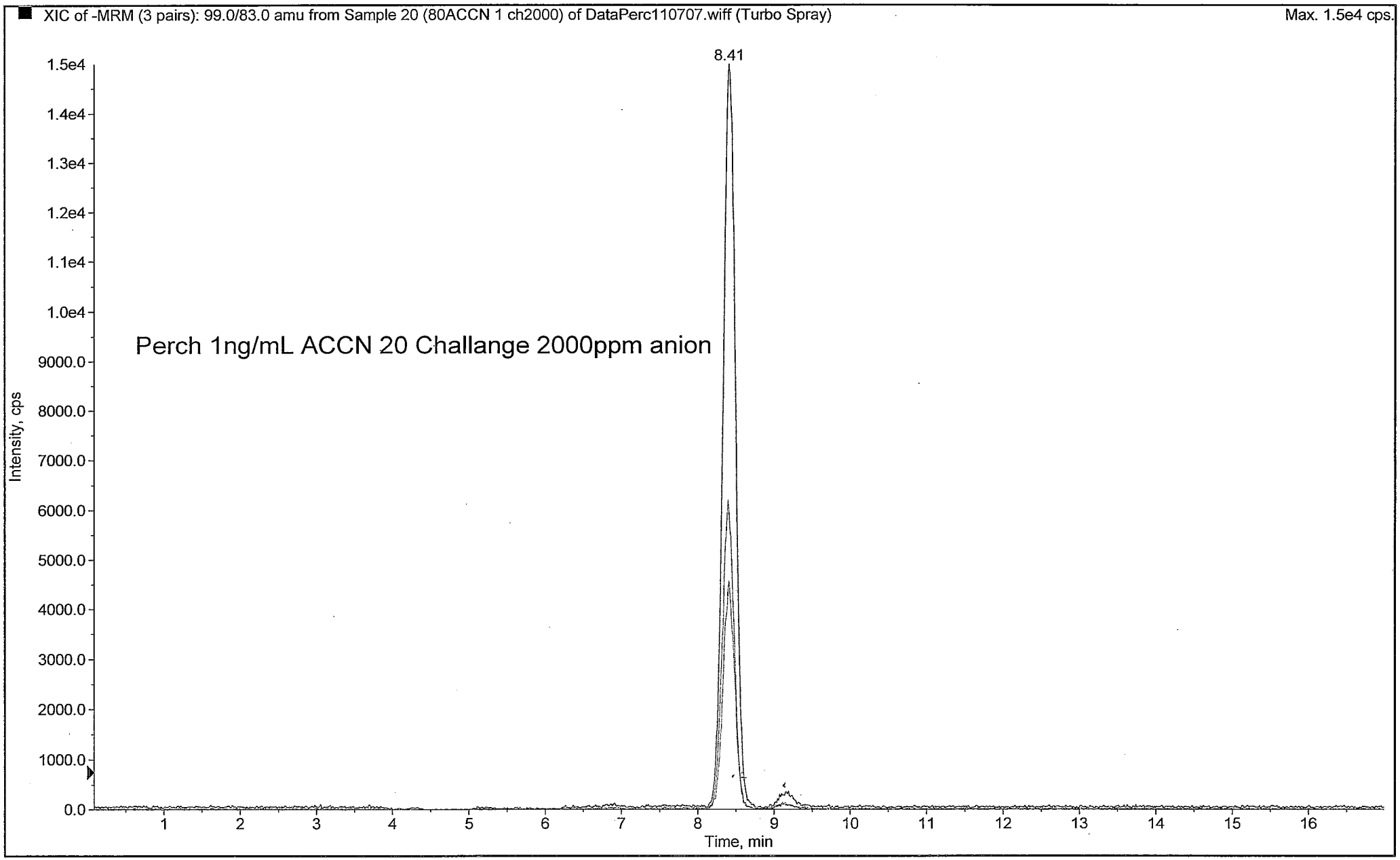
Printing Date: Thursday, November 08, 2007



Operator: jefferie
Printing Time: 12:14:44 PM
Printing Date: Thursday, November 08, 2007



Operator: jefferie
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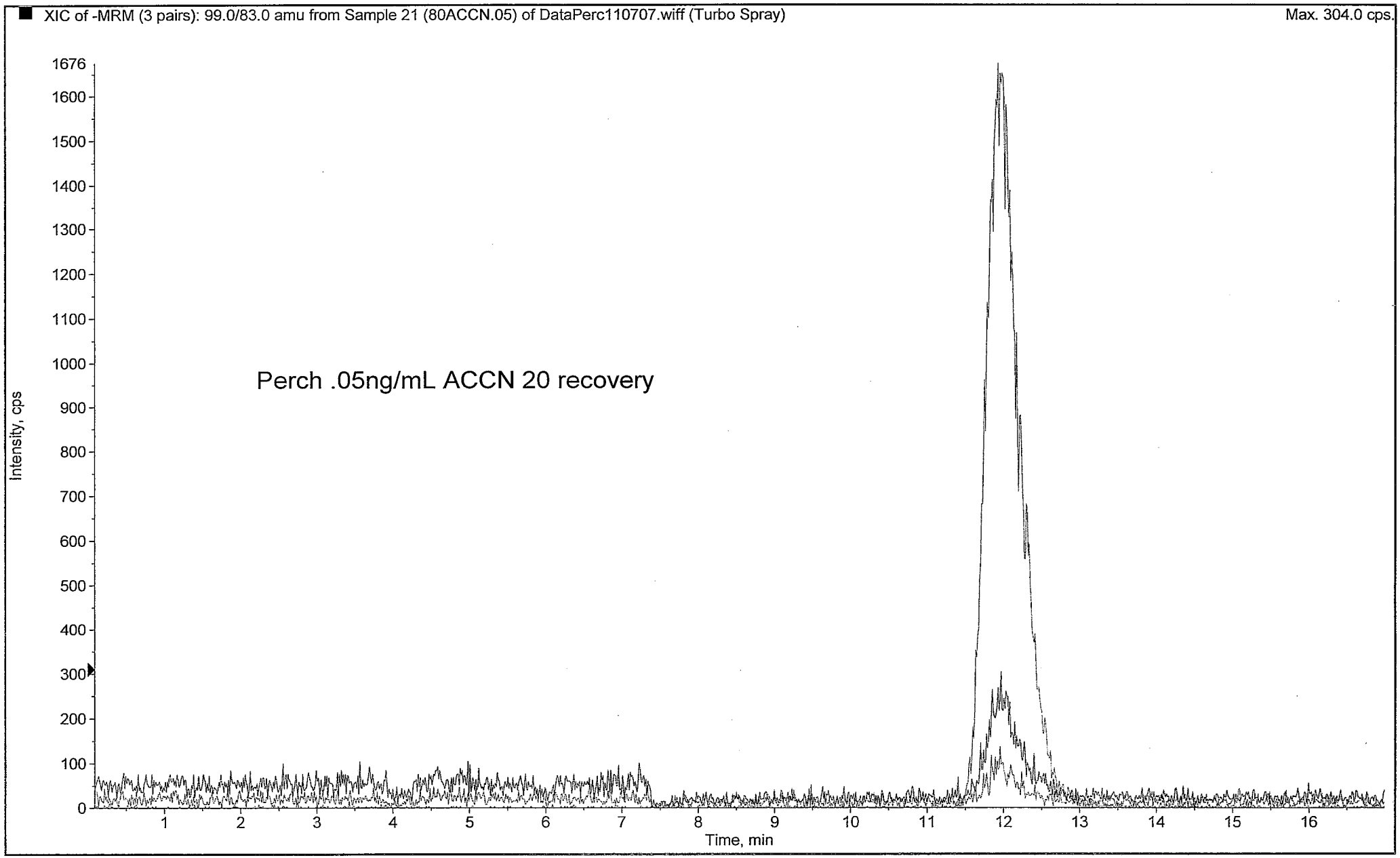
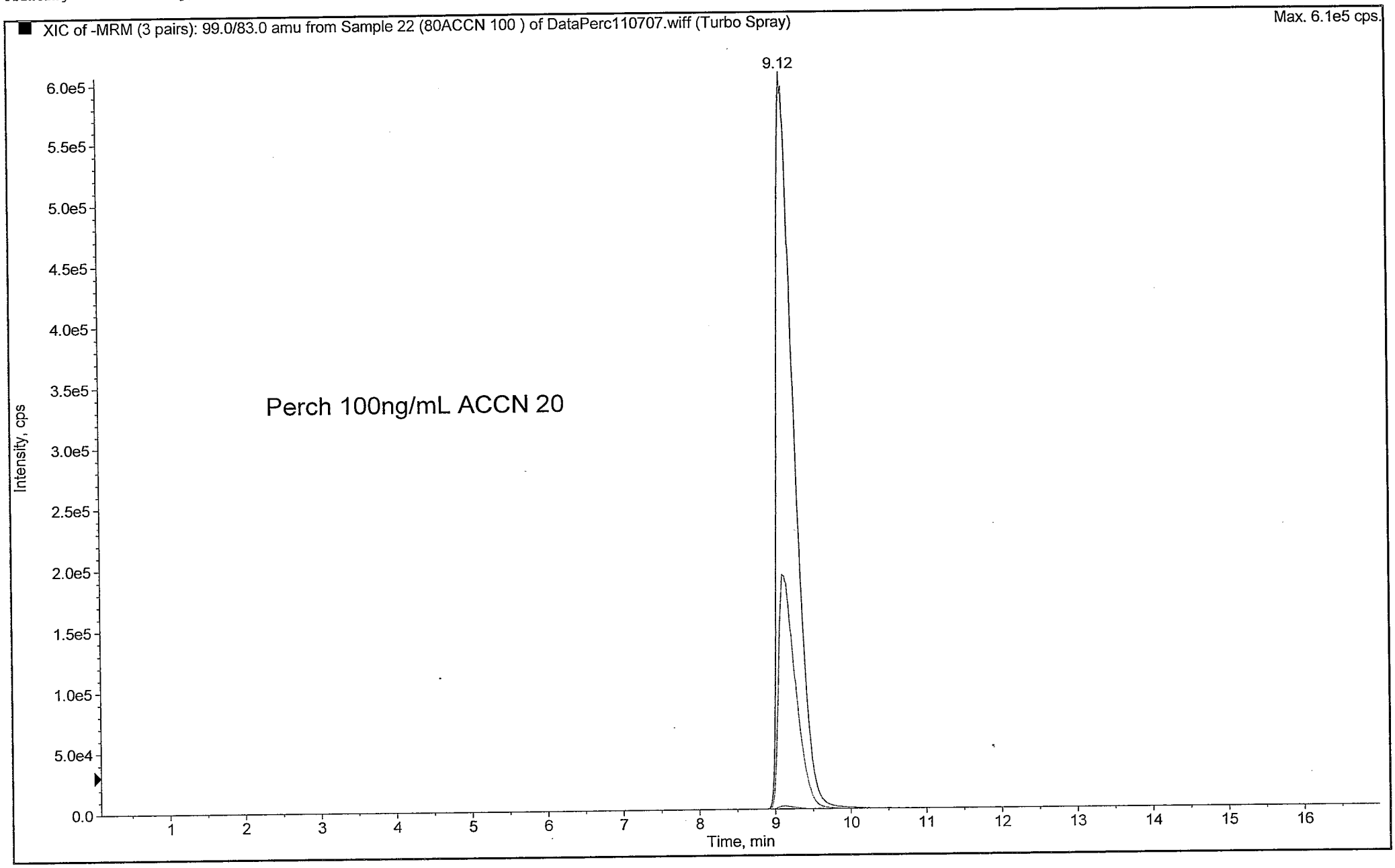


Operator: jefferie
Printing Time: 12:16:48 PM
Printing Date: Thursday, November 08, 2007



Operator: jefferie
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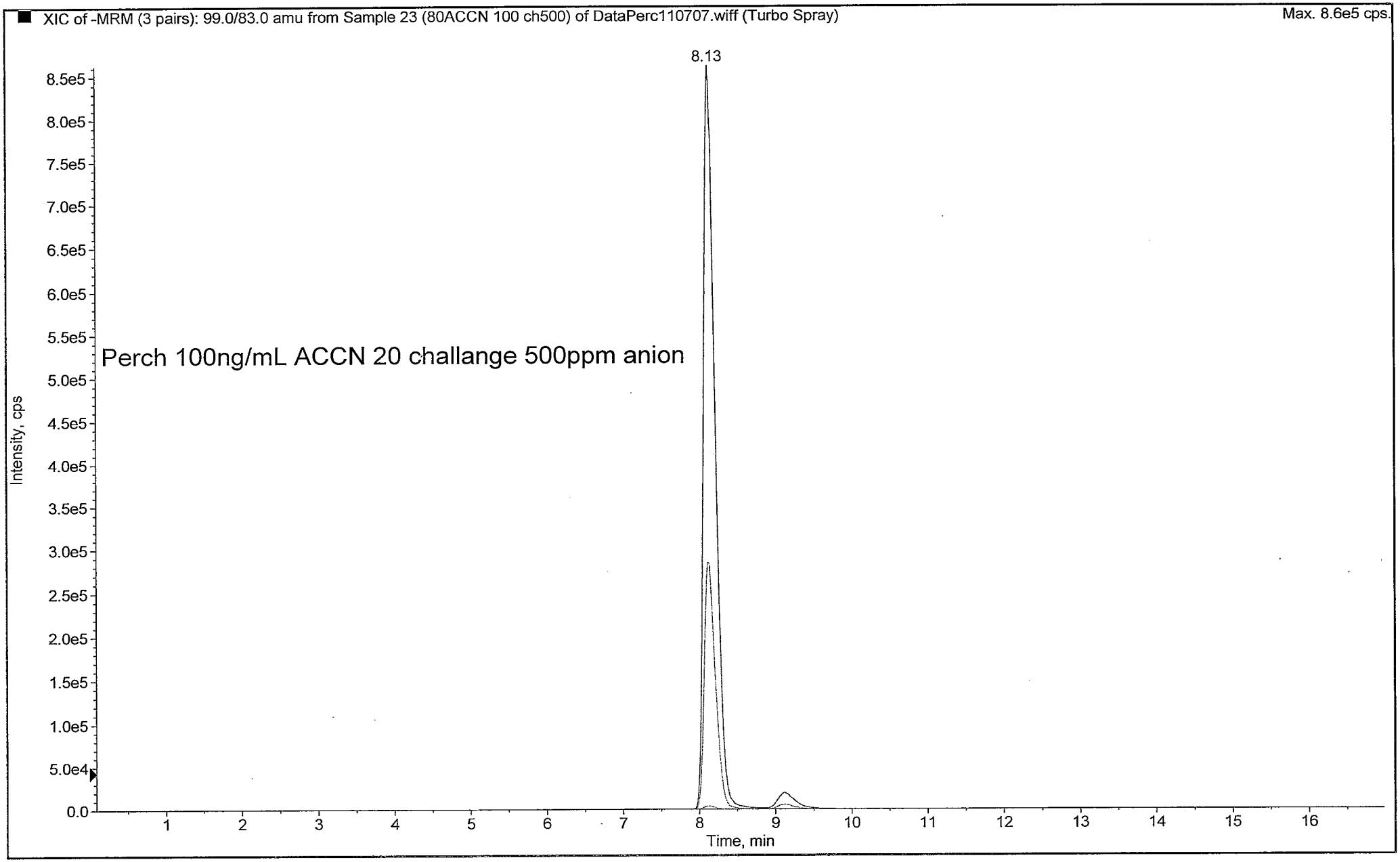

Operator: jefferie
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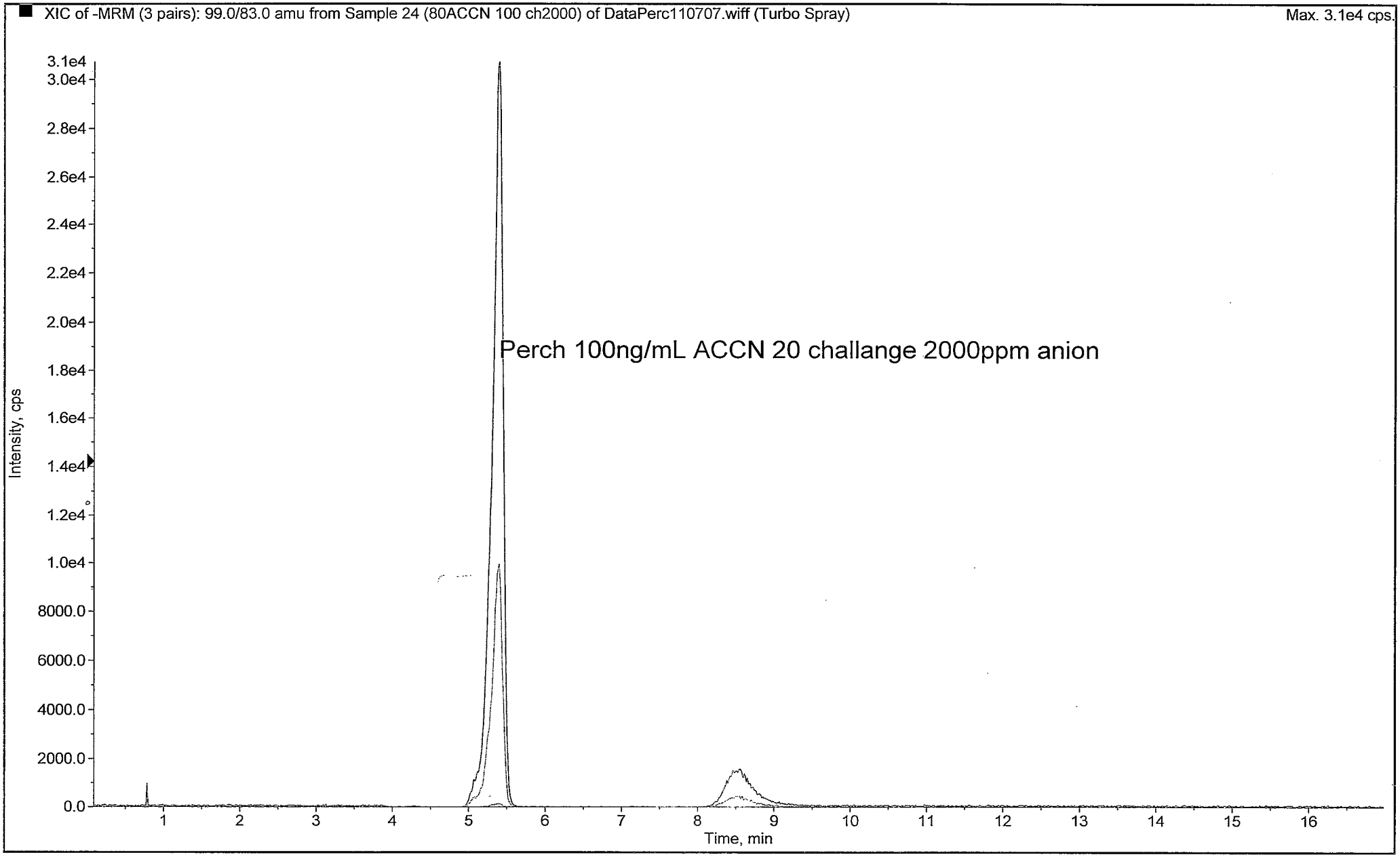

Operator: jefferie
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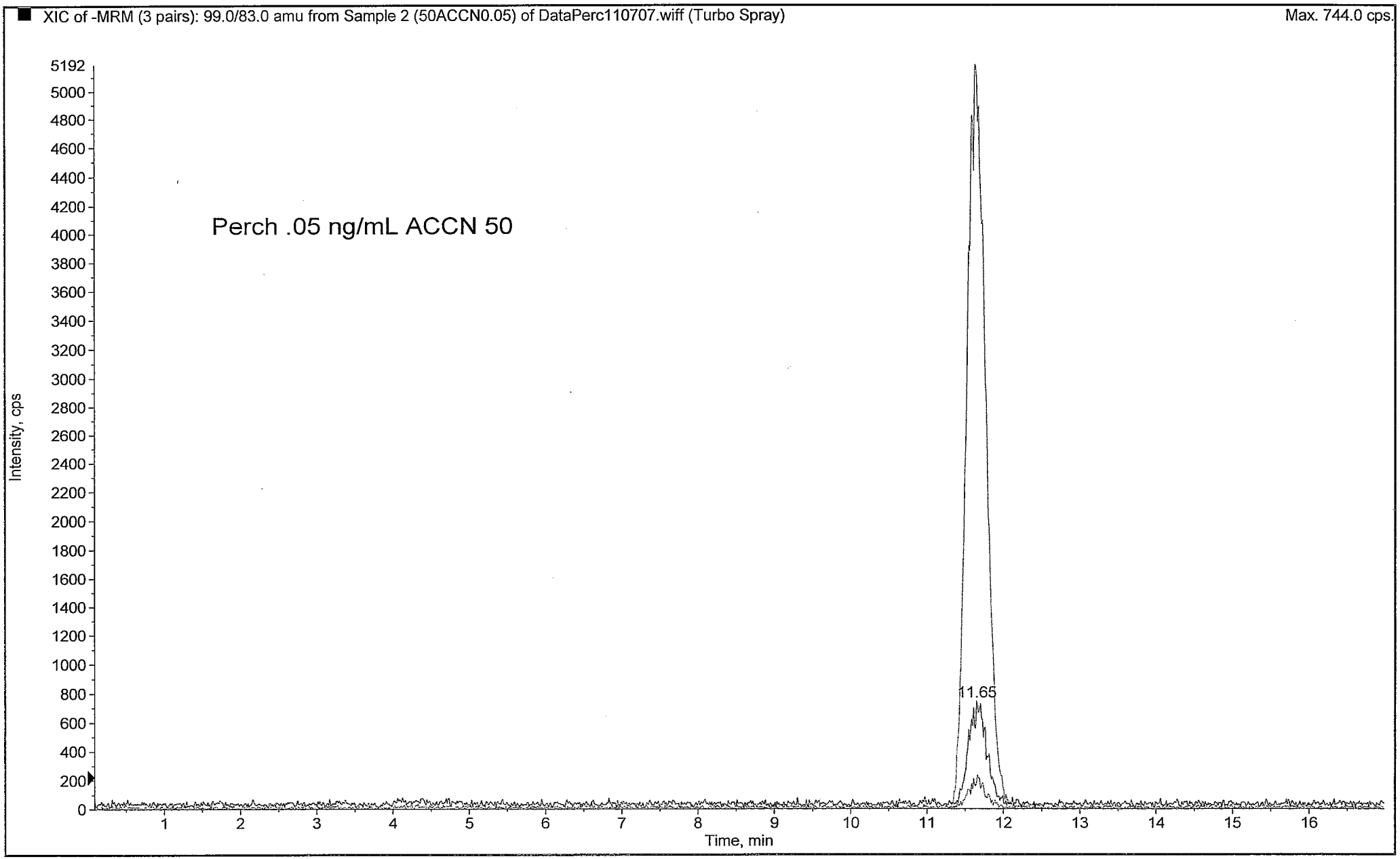

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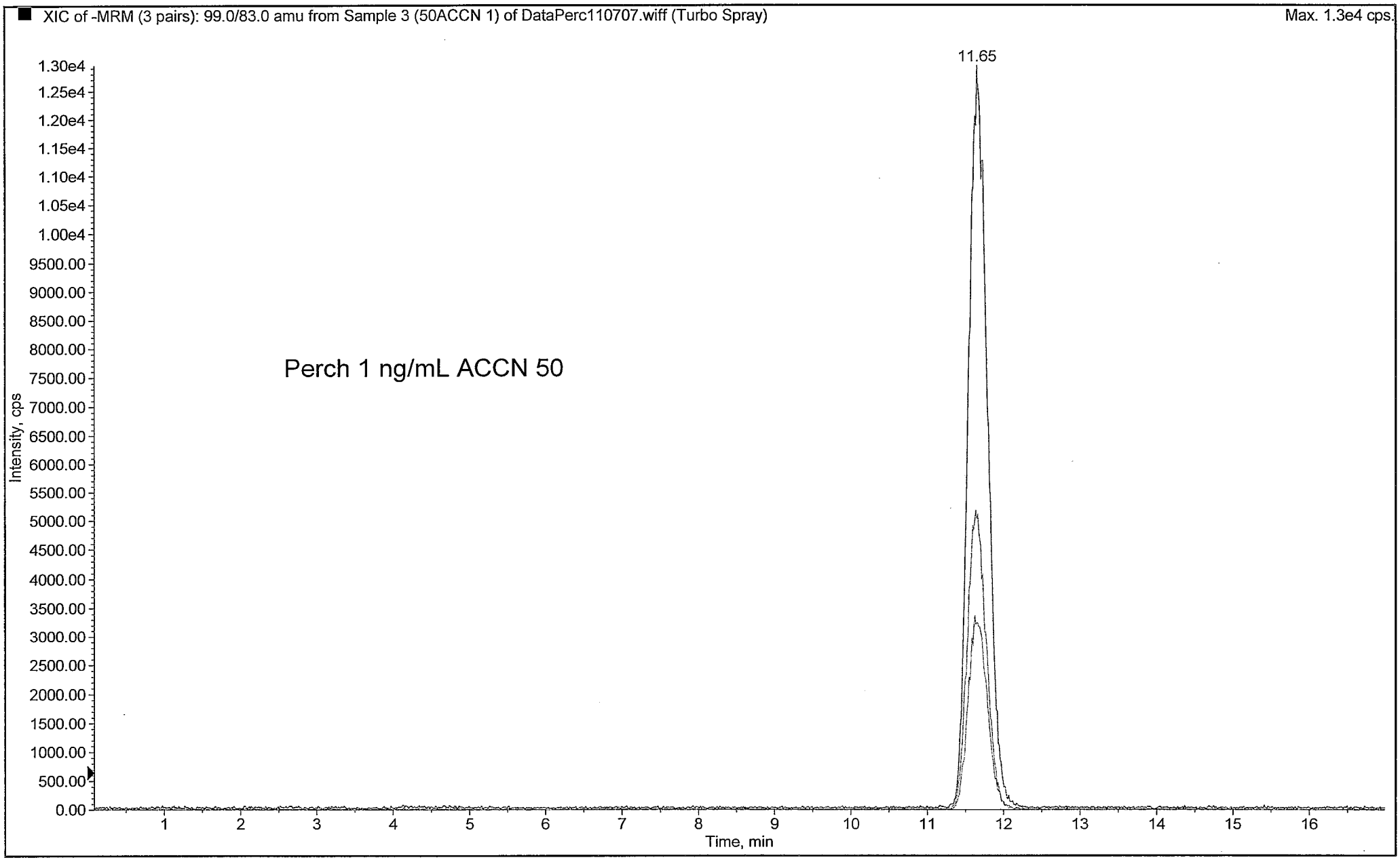

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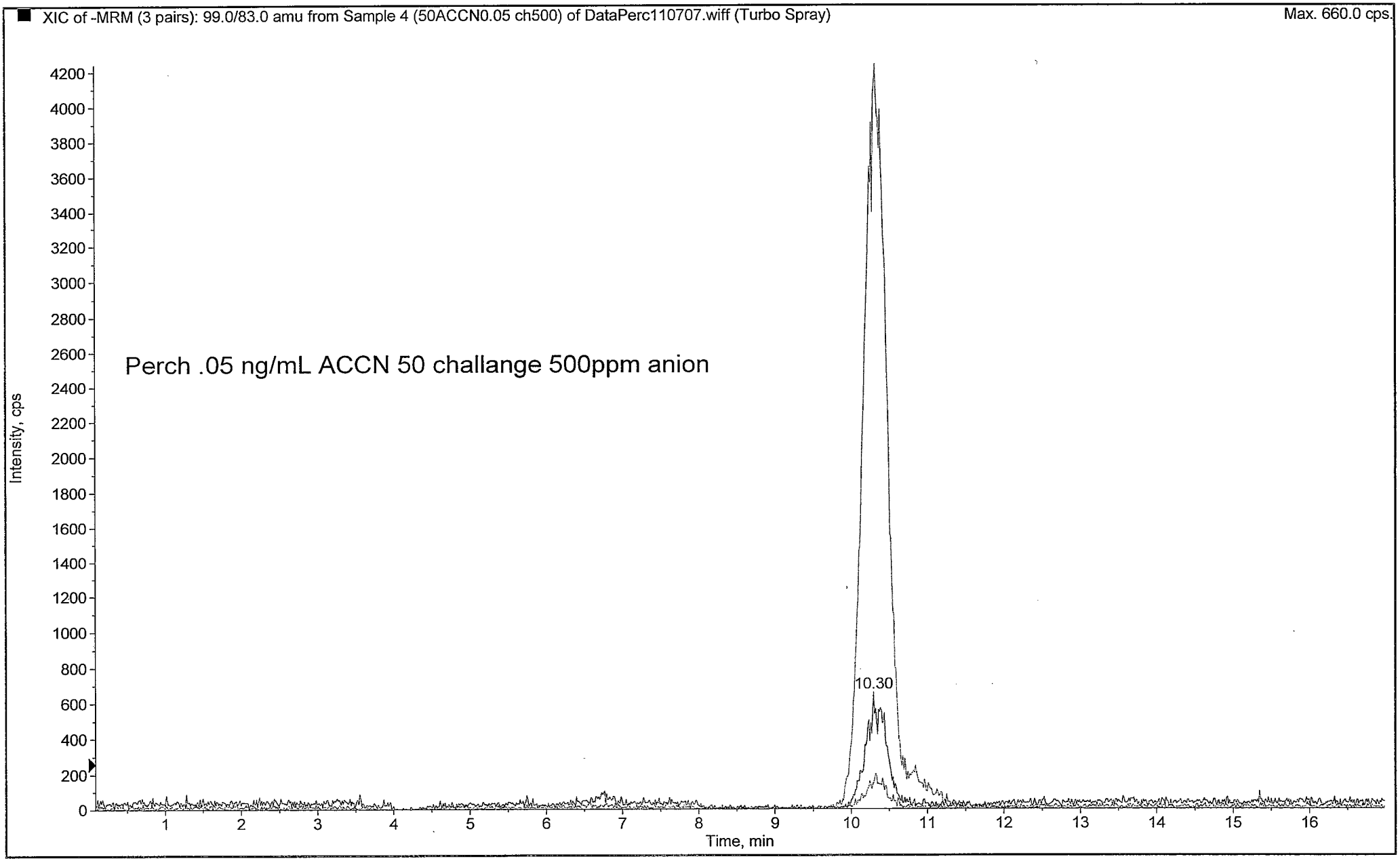

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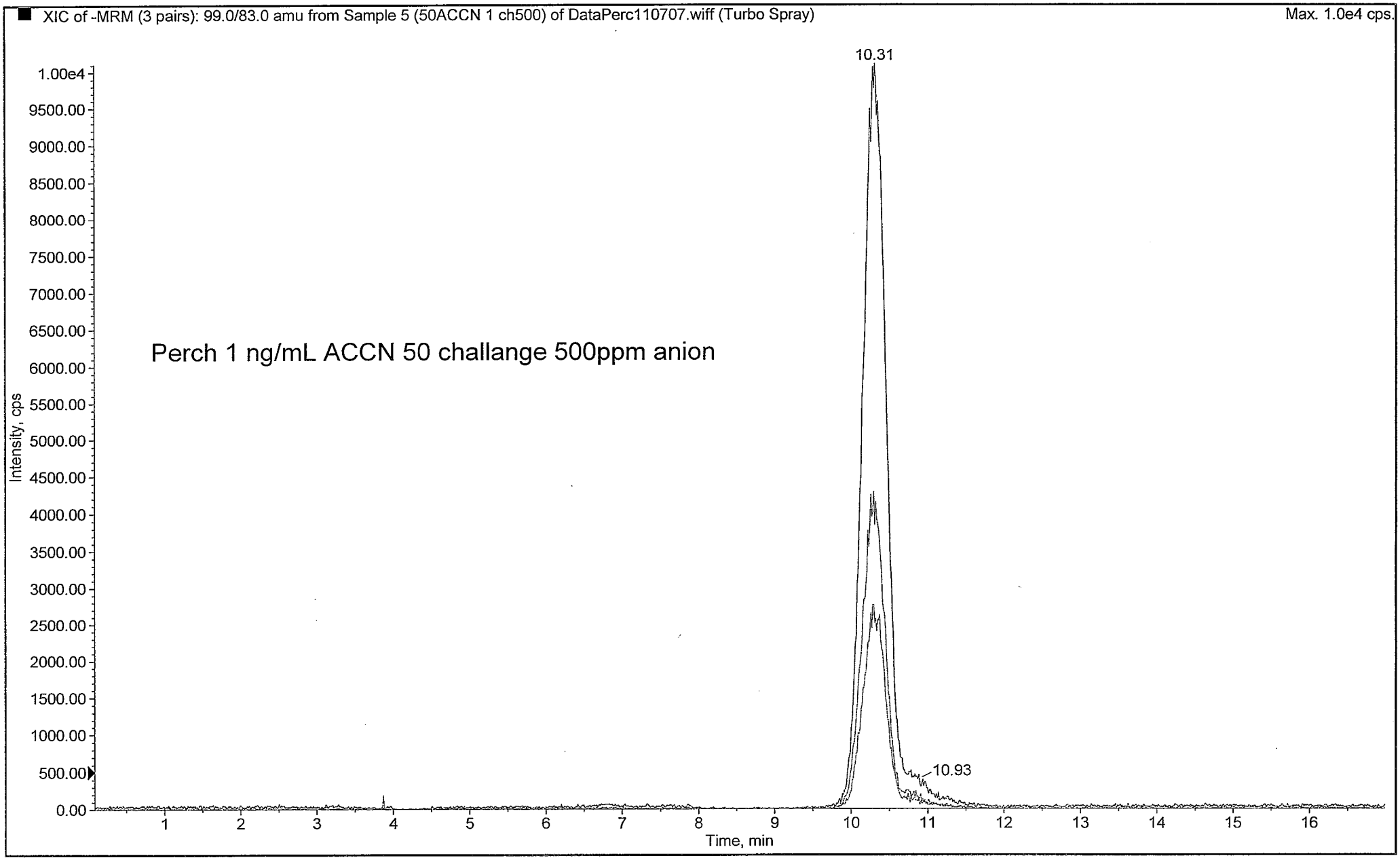

Operator: jefferie
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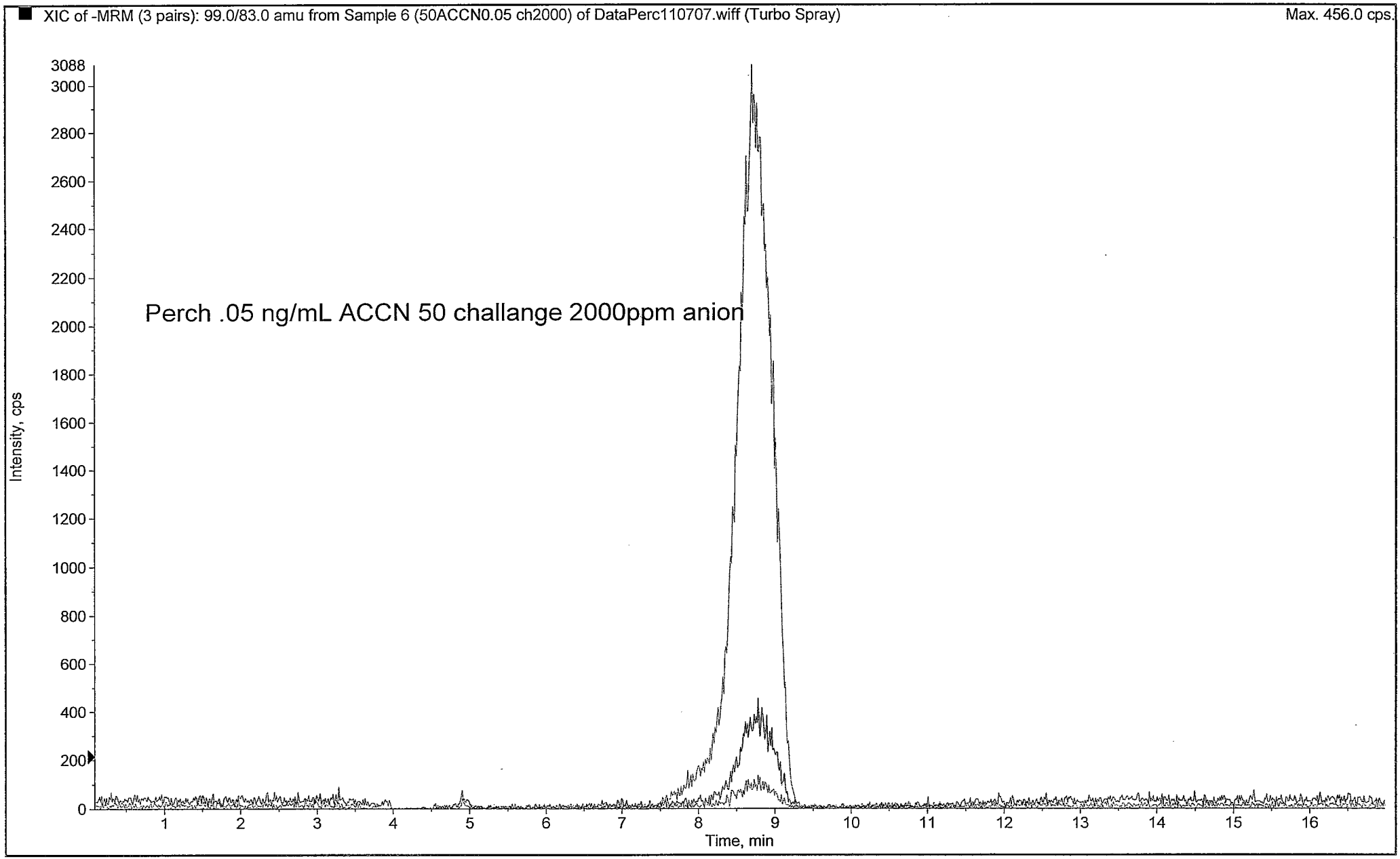

Operator: jefferie
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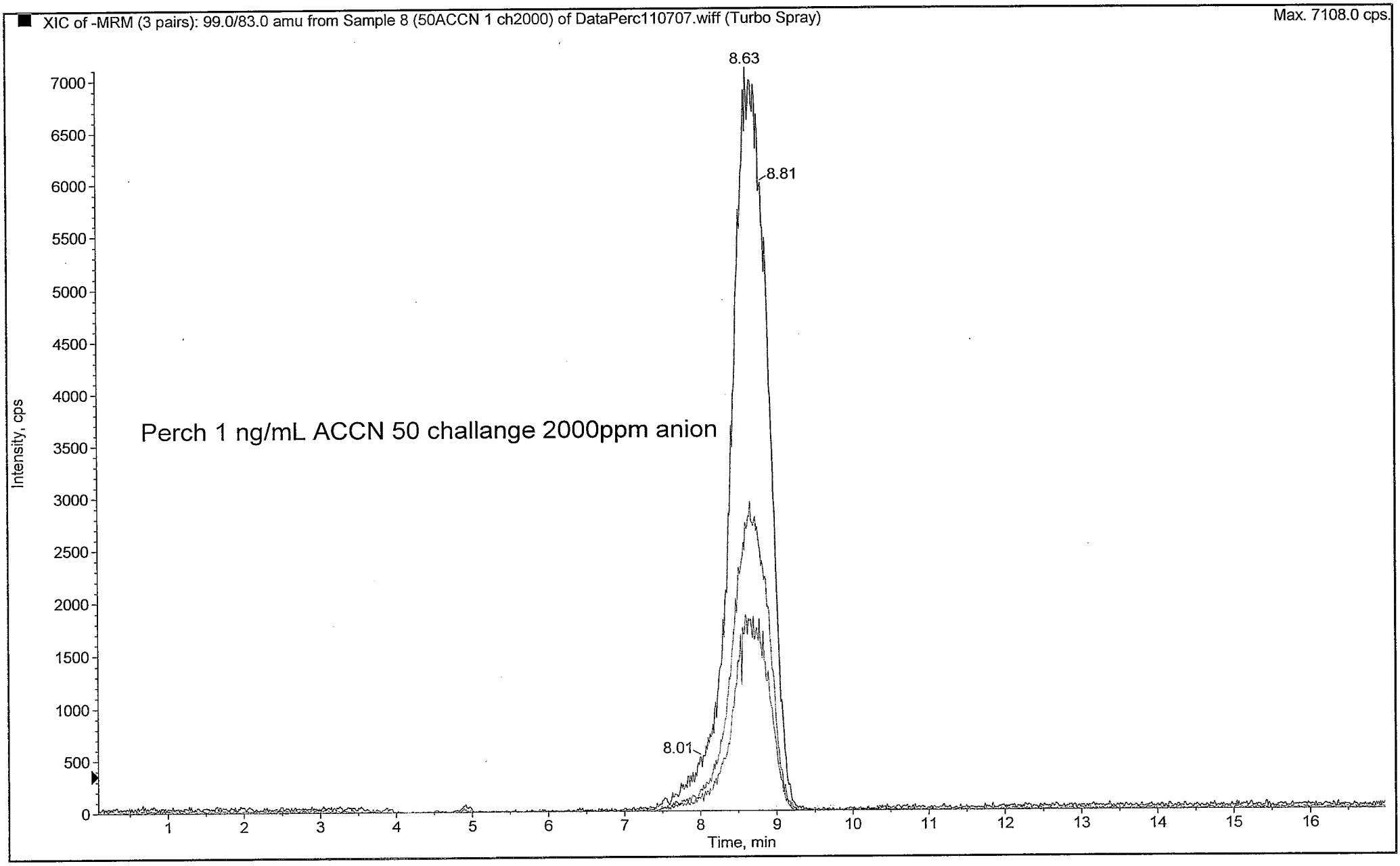
Printing Date: Thursday, November 08, 2007



Operator: jefferie
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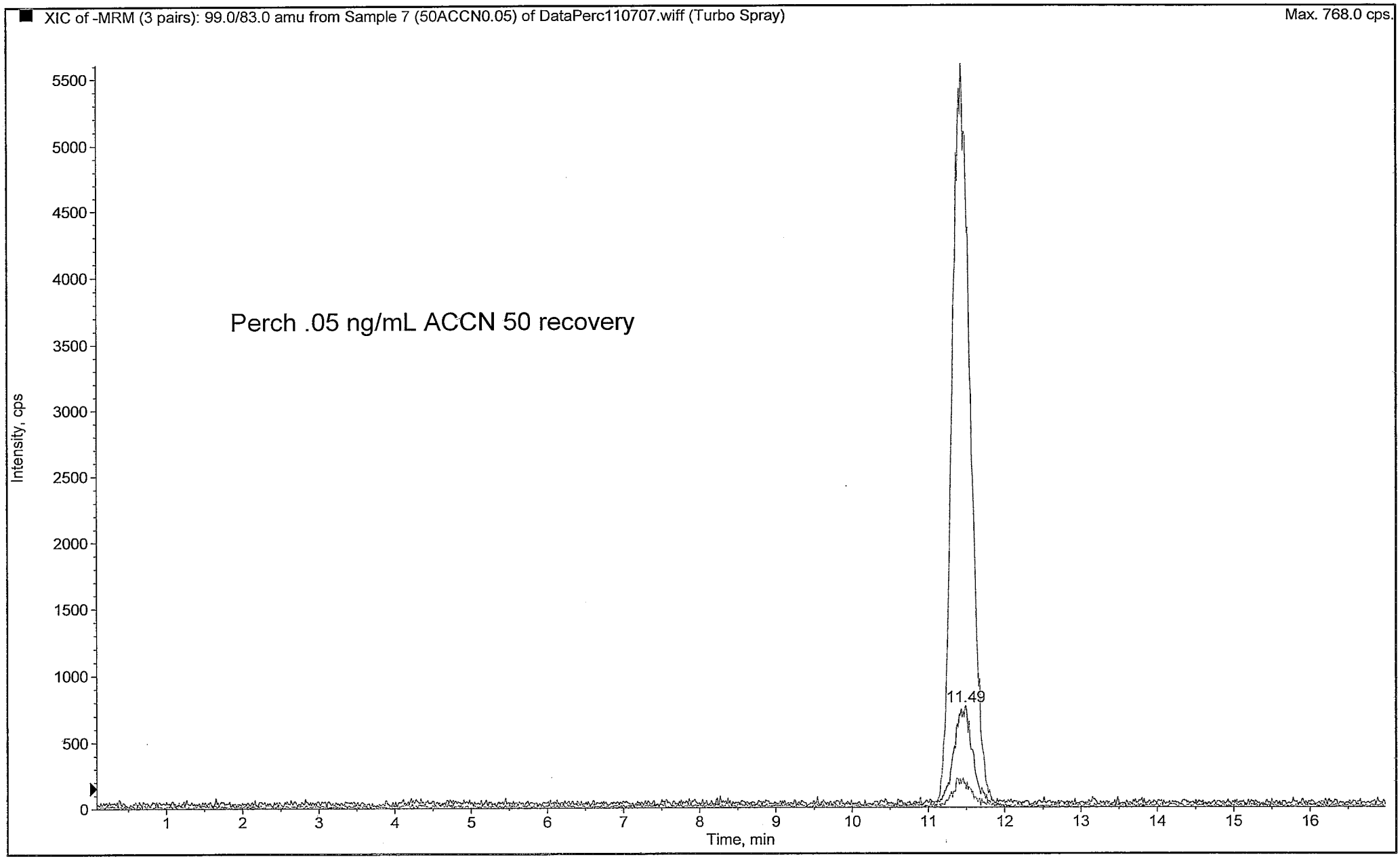
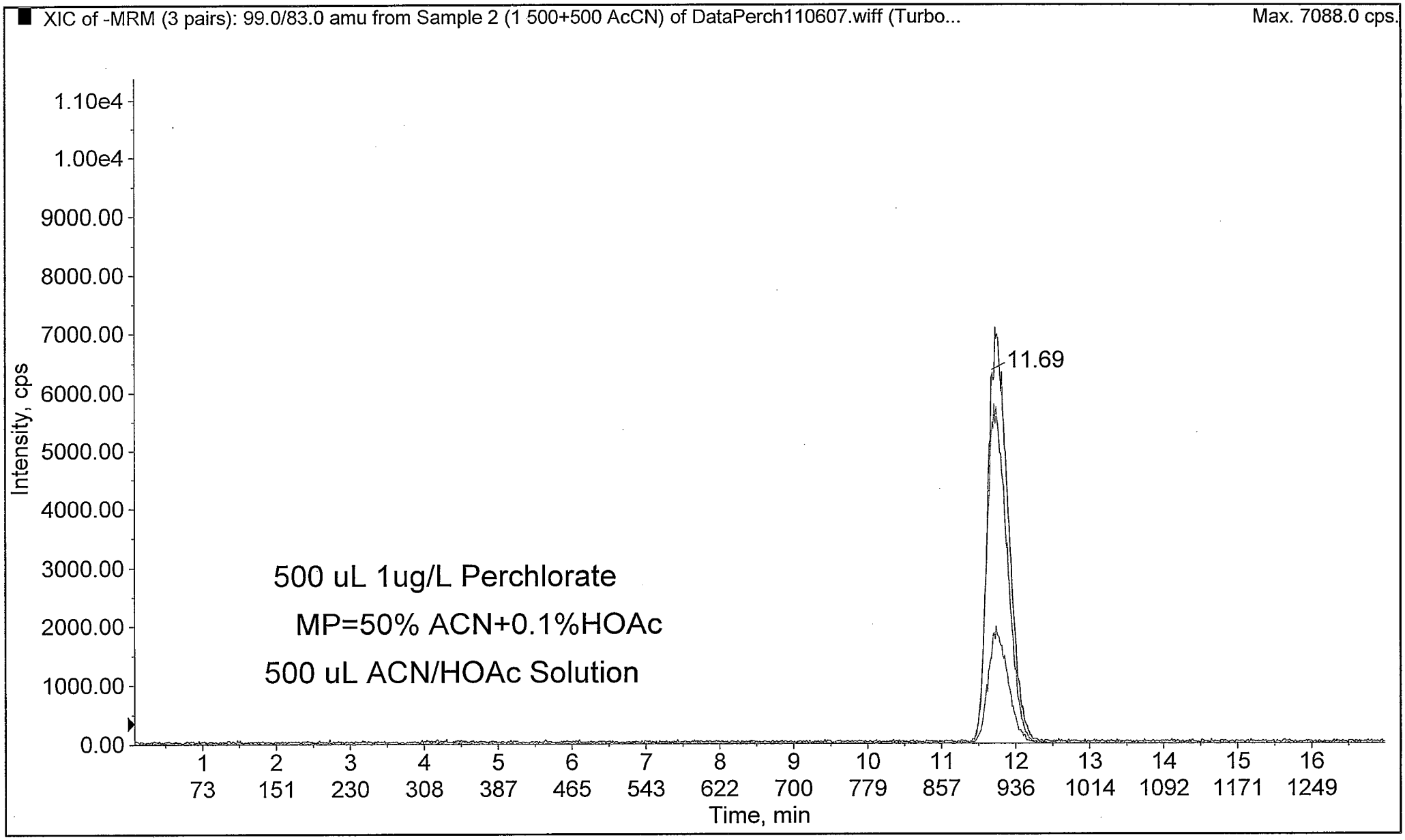
Printing Date: Thursday, November 08, 2007

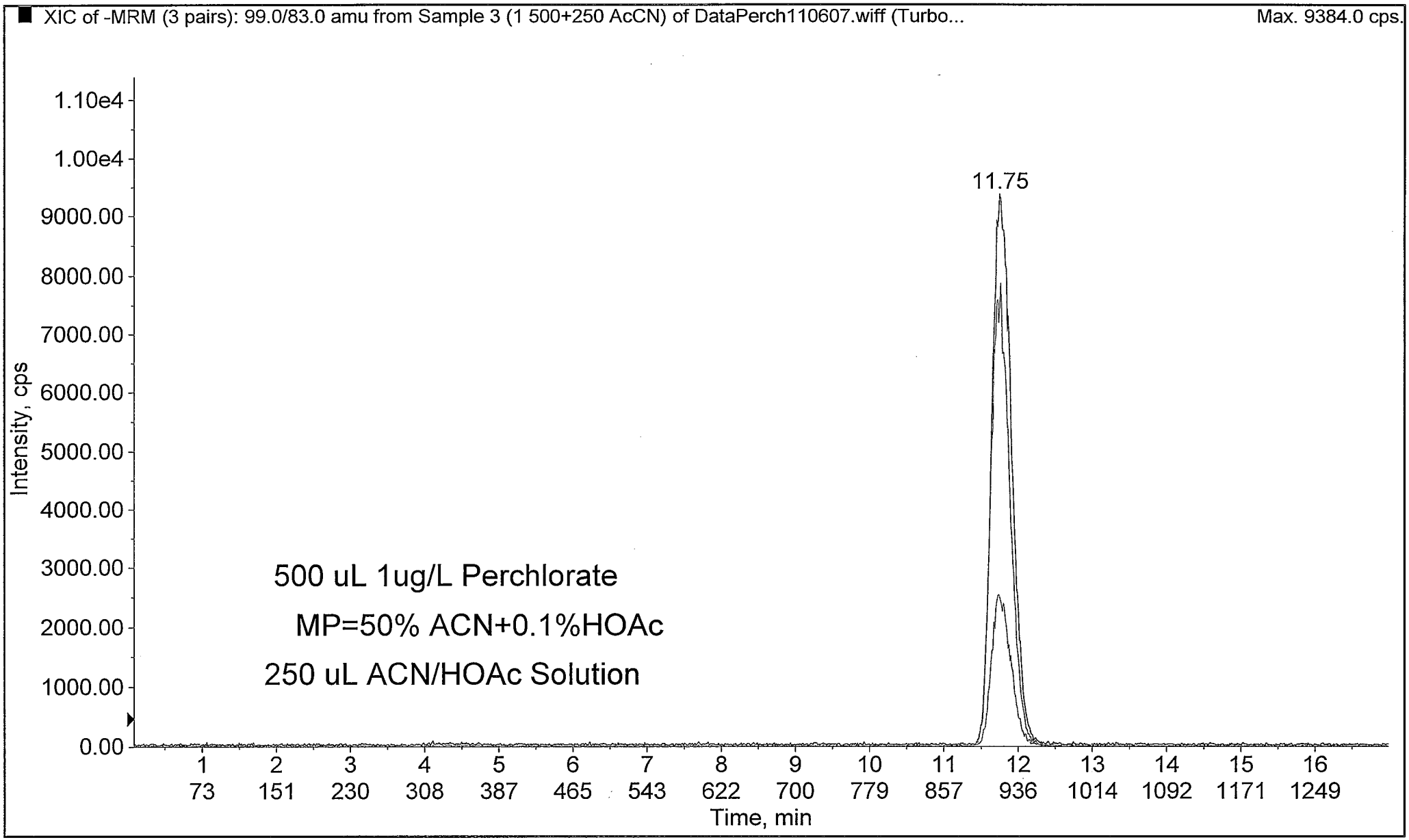


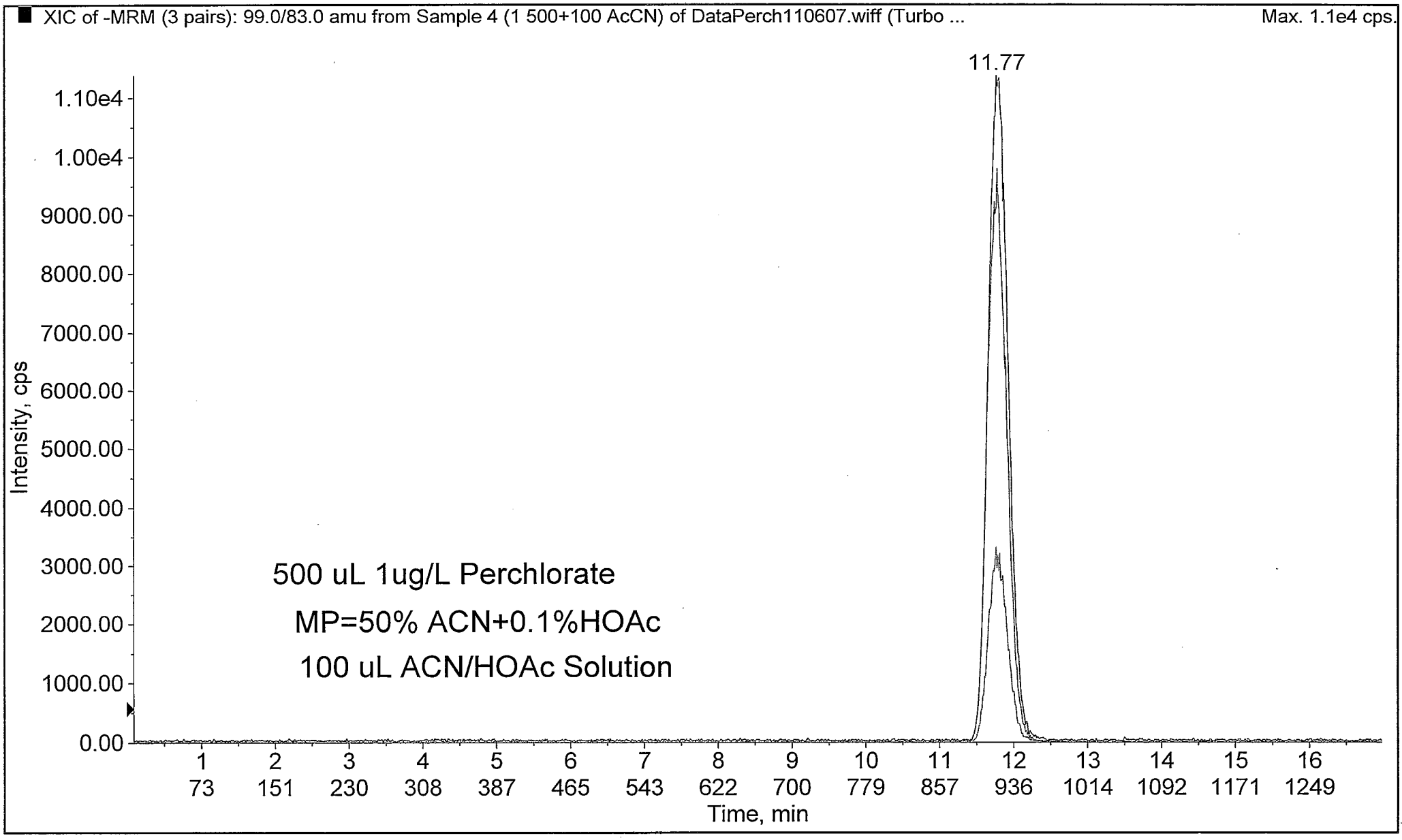
Appendix III

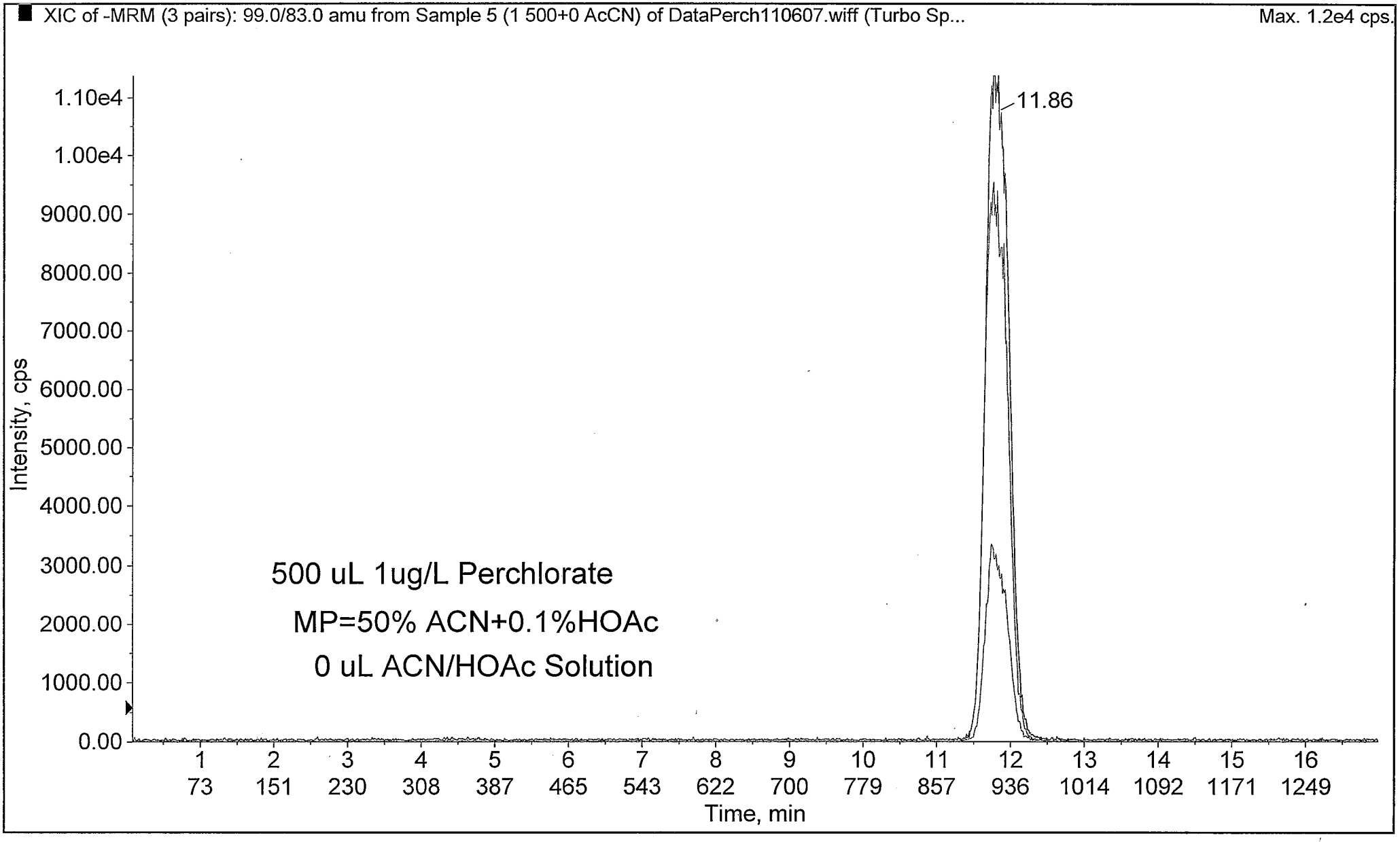
Matching Mobile Phase Composition in Sample Extracts for Peak Share Optimization

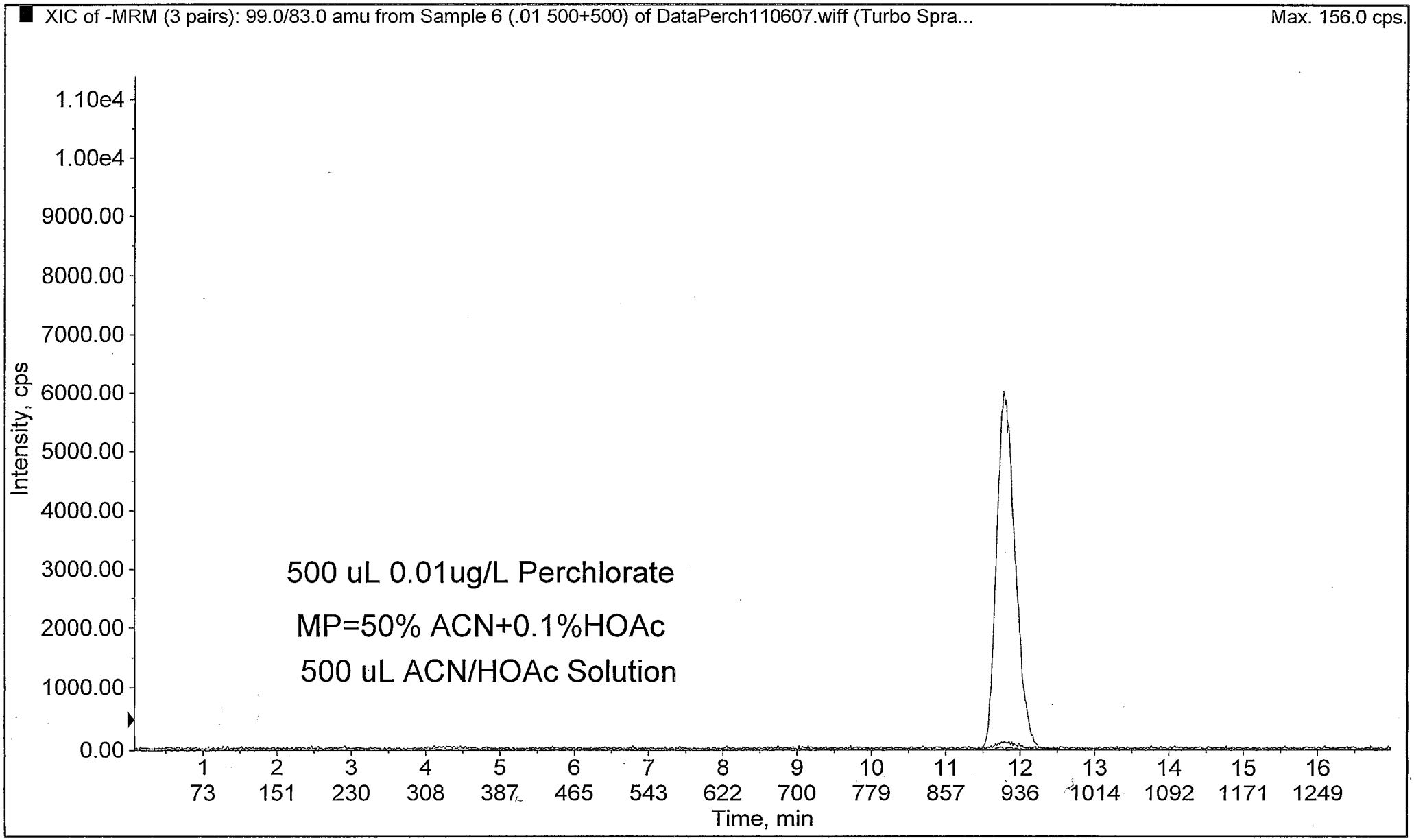
(ACN/HOAc addition to sample)

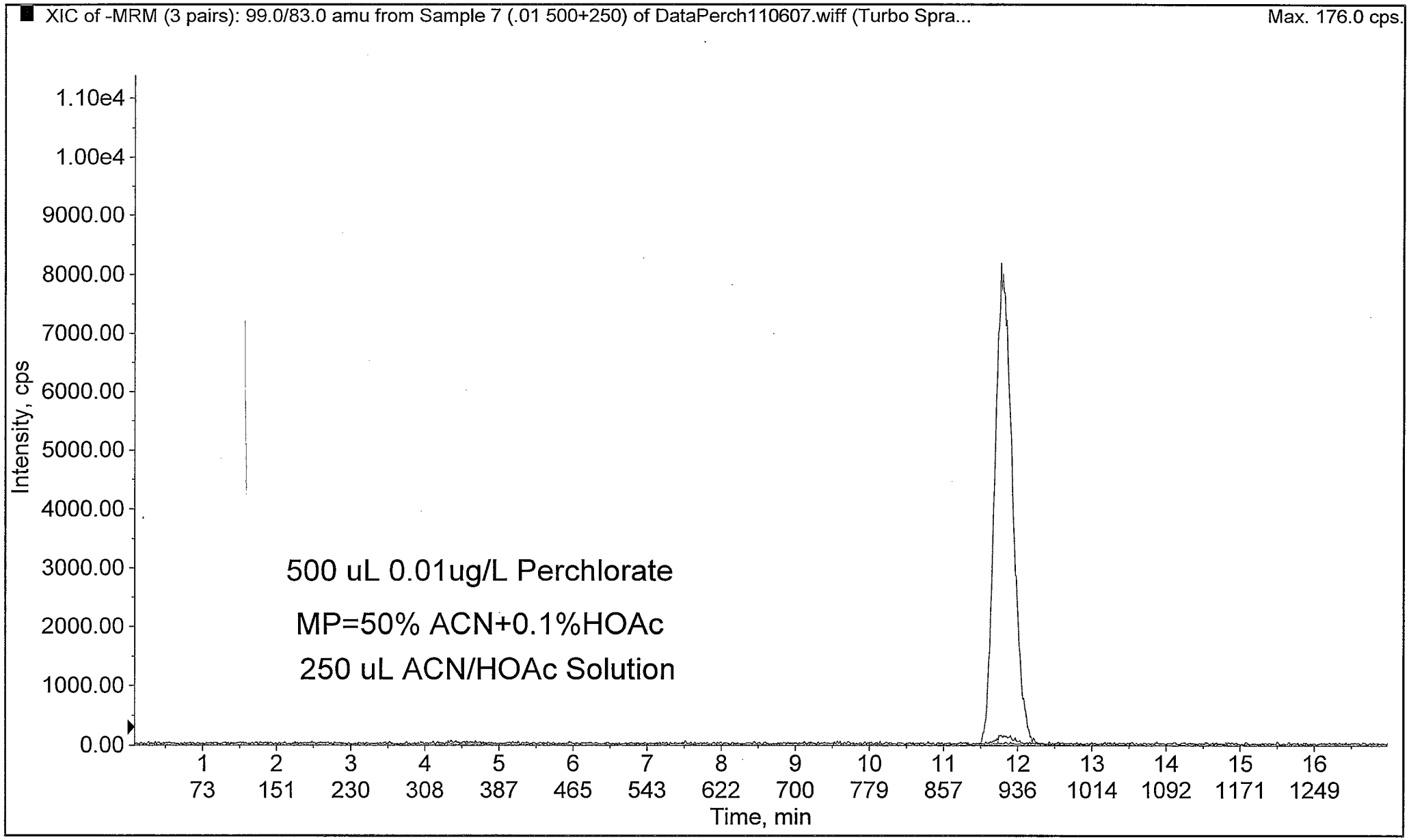

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 2 (1 500+500 AcCN) of DataPerch110607.wiff (Turbo...
Max. 7088.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
11.69
500 uL 1ug/L Perchlorate
MP=50% ACN+0.1%HOAc
500 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

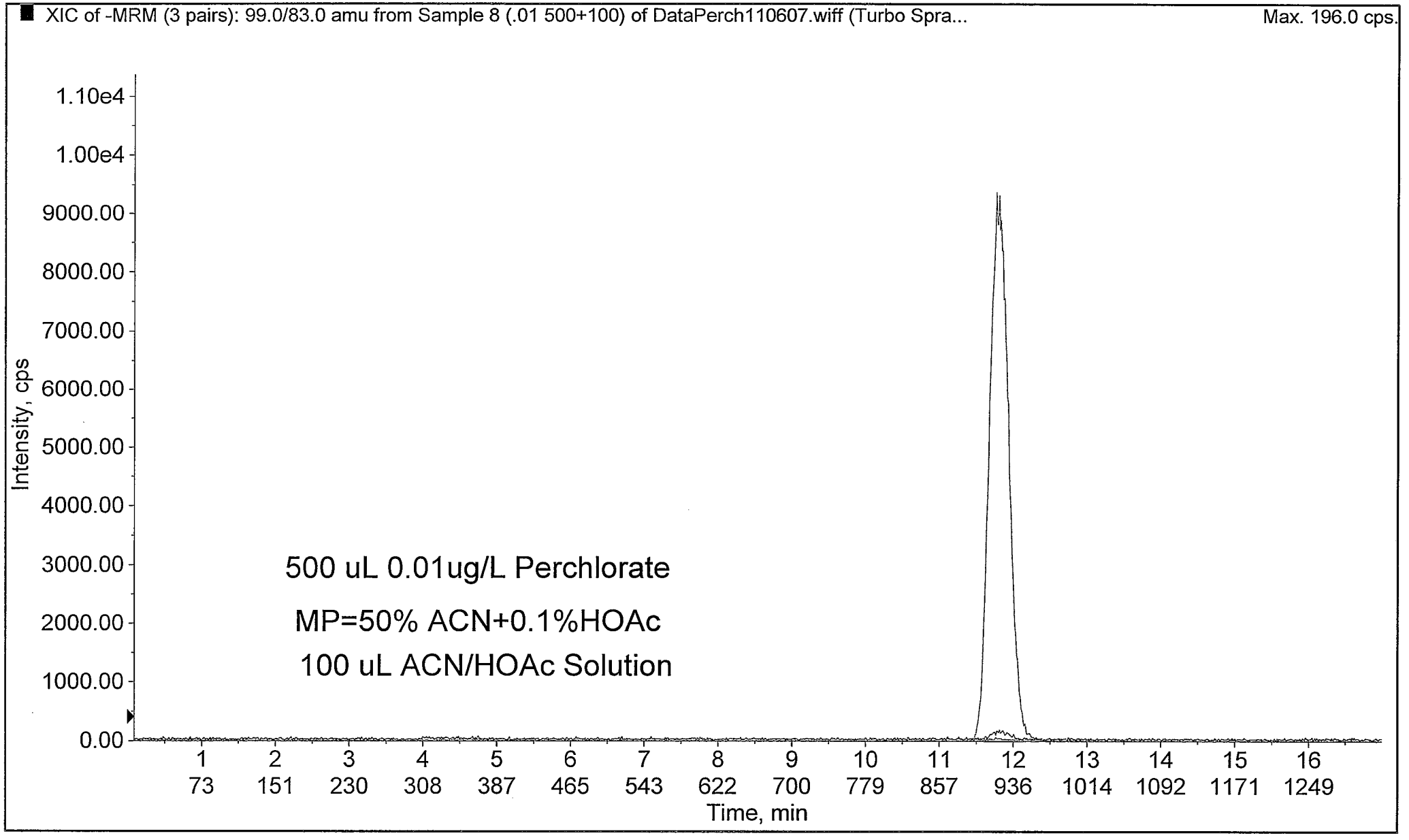

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 3 (1 500+250 AcCN) of DataPerch110607.wiff (Turbo...
Max. 9384.0 cps.
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
Intensity, cps
11.75
500 uL 1ug/L Perchlorate
MP=50% ACN+0.1%HOAc
250 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

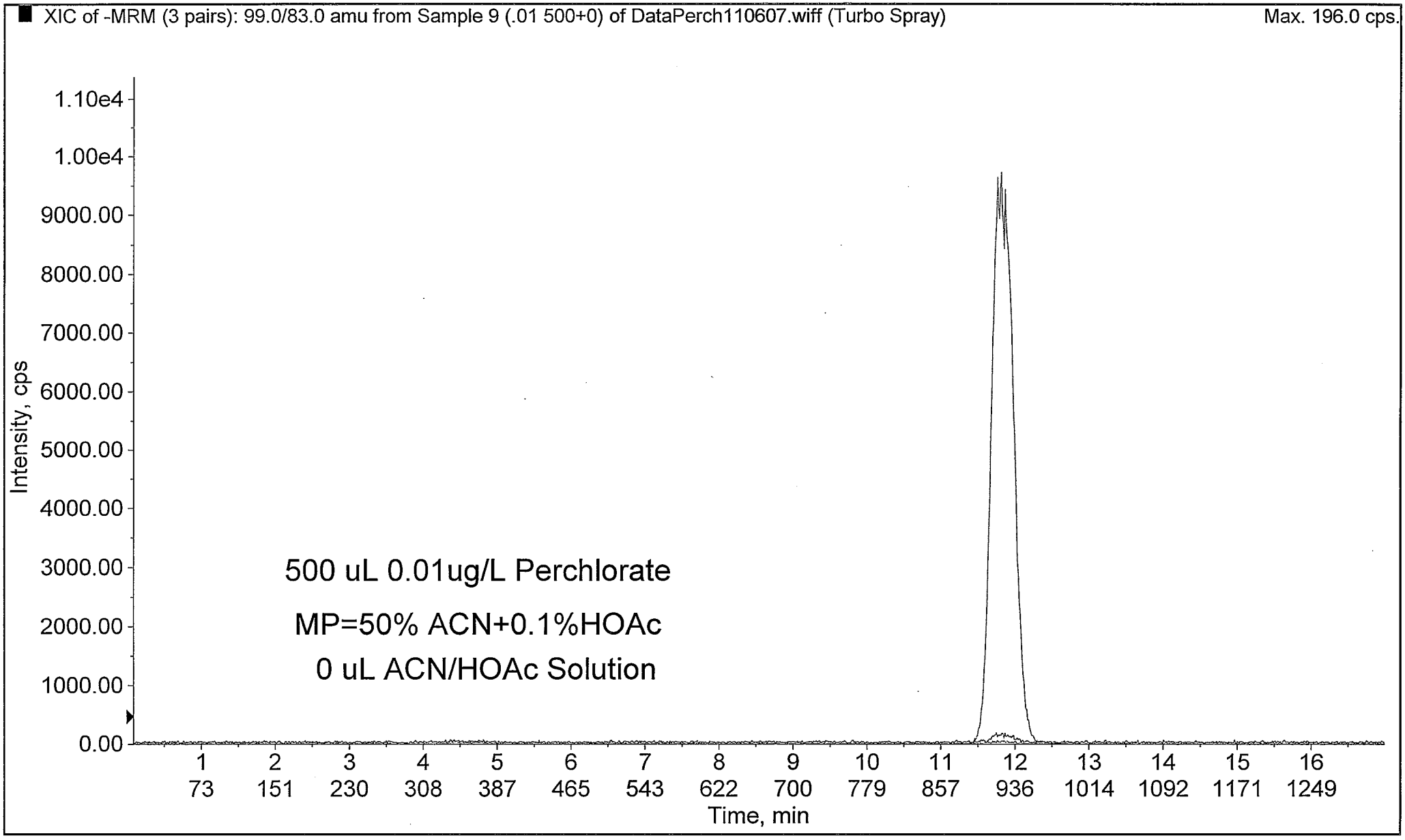

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 4 (1 500+100 AcCN) of DataPerch110607.wiff (Turbo ...
Max. 1.1e4 cps.
11.77
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
Intensity, cps
500 uL 1ug/L Perchlorate
MP=50% ACN+0.1%HOAc
100 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

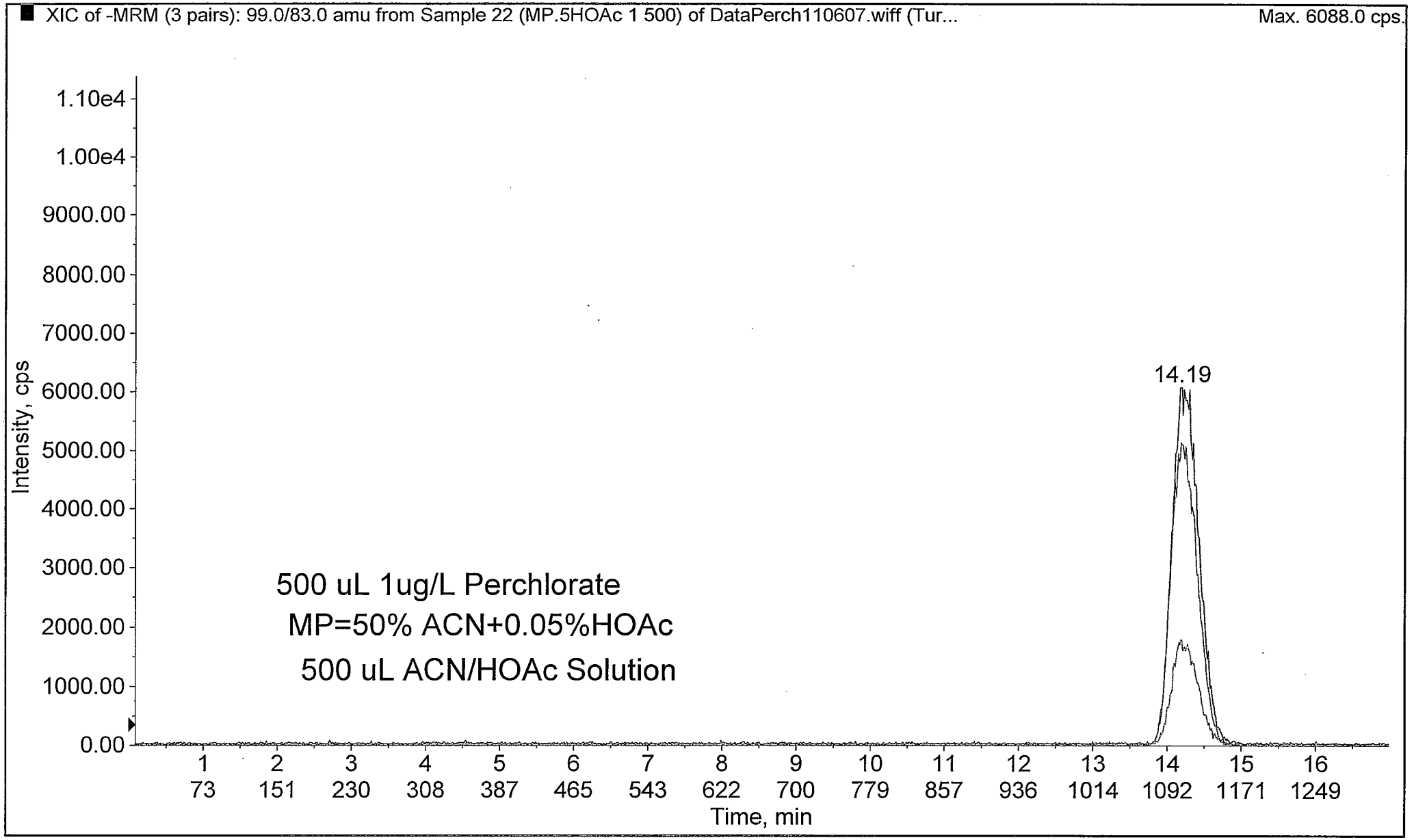

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 5 (1 500+0 AcCN) of DataPerch110607.wiff (Turbo Sp...
Max. 1.2e4 cps.
11.86
500 uL 1ug/L Perchlorate
MP=50% ACN+0.1%HOAc
0 uL ACN/HOAc Solution
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

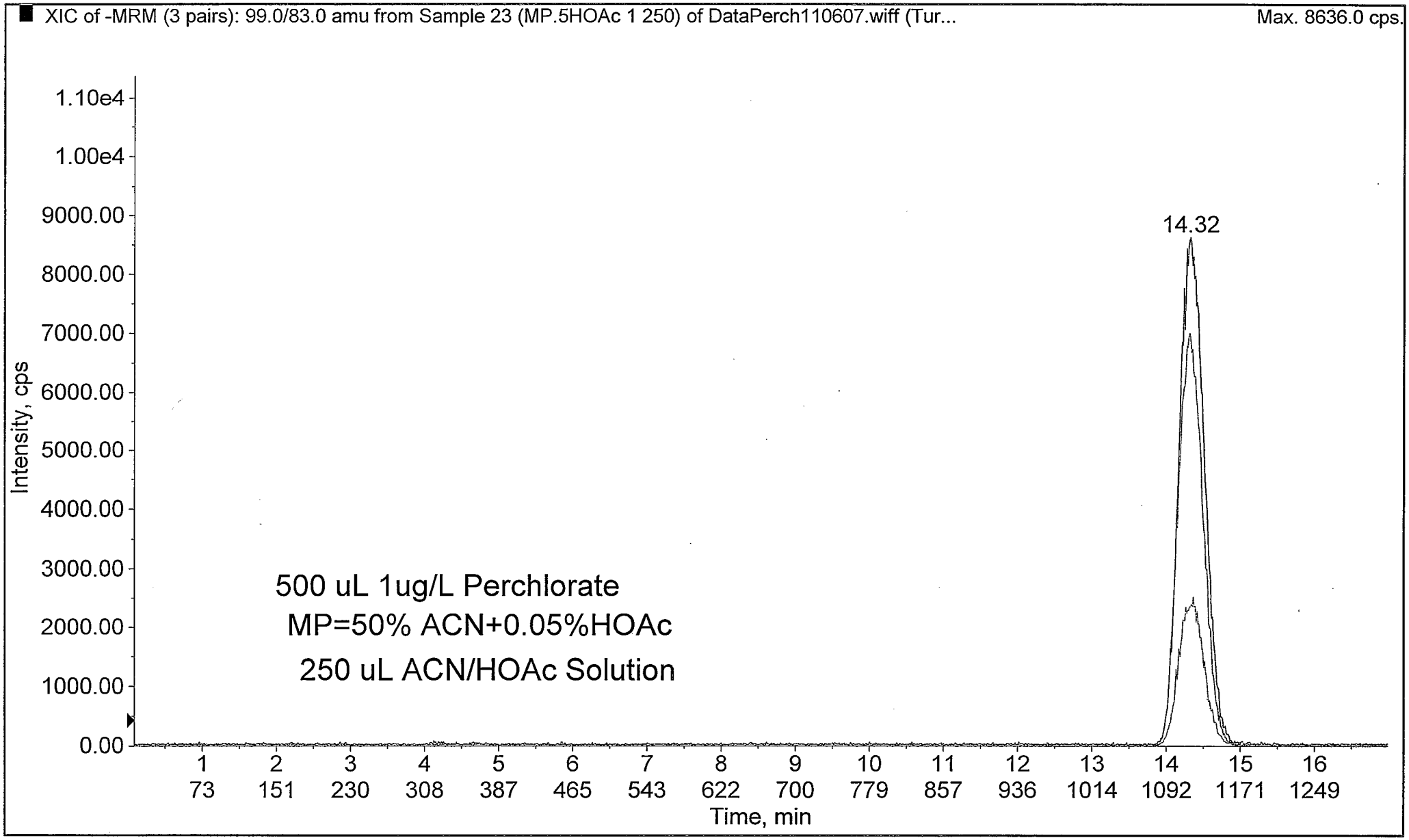

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 6 (.01 500+500) of DataPerch110607.wiff (Turbo Spra...
Max. 156.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL 0.01ug/L Perchlorate
MP=50% ACN+0.1%HOAc
500 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

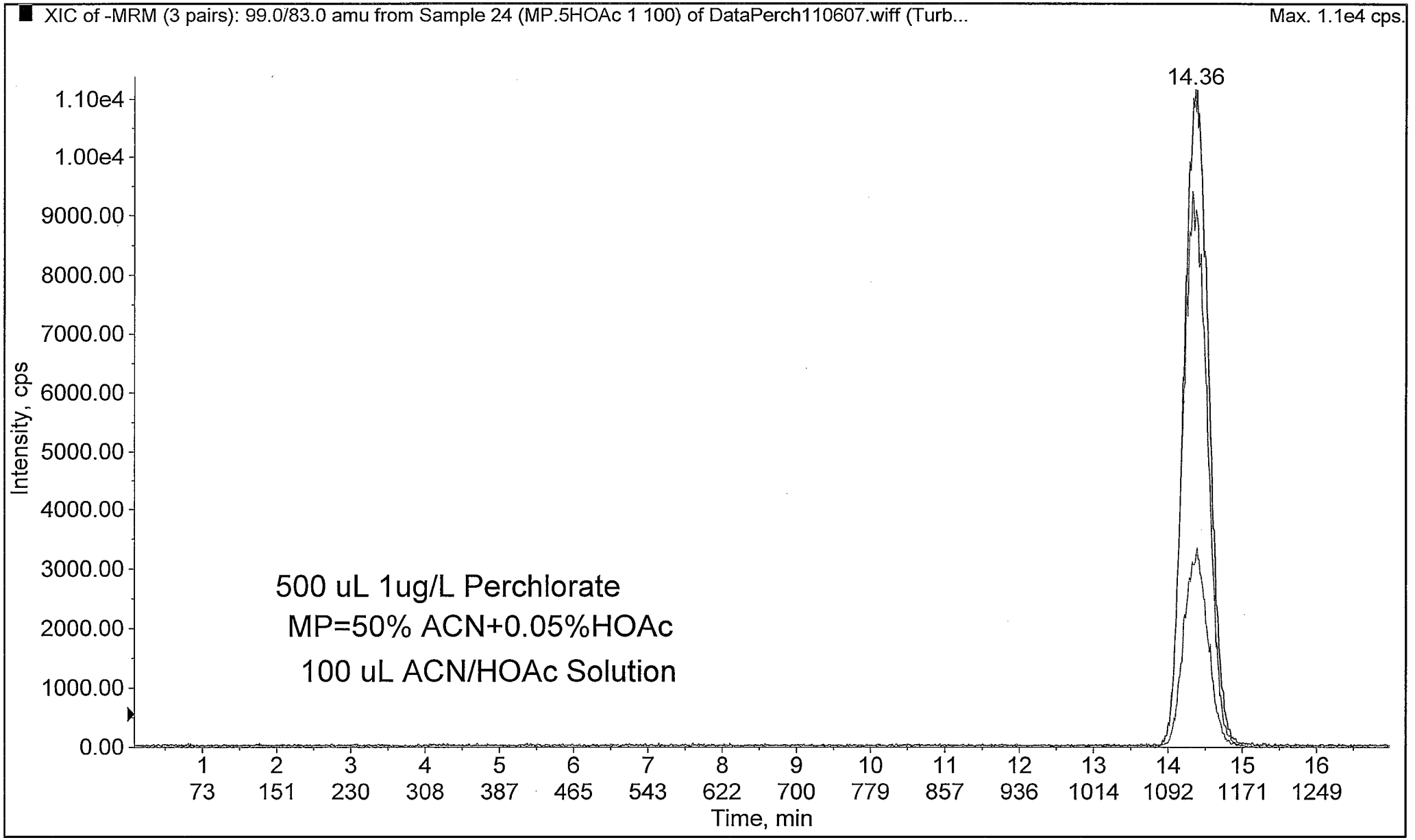

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 7 (.01 500+250) of DataPerch110607.wiff (Turbo Spra...
Max. 176.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL 0.01ug/L Perchlorate
MP=50% ACN+0.1%HOAc
250 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

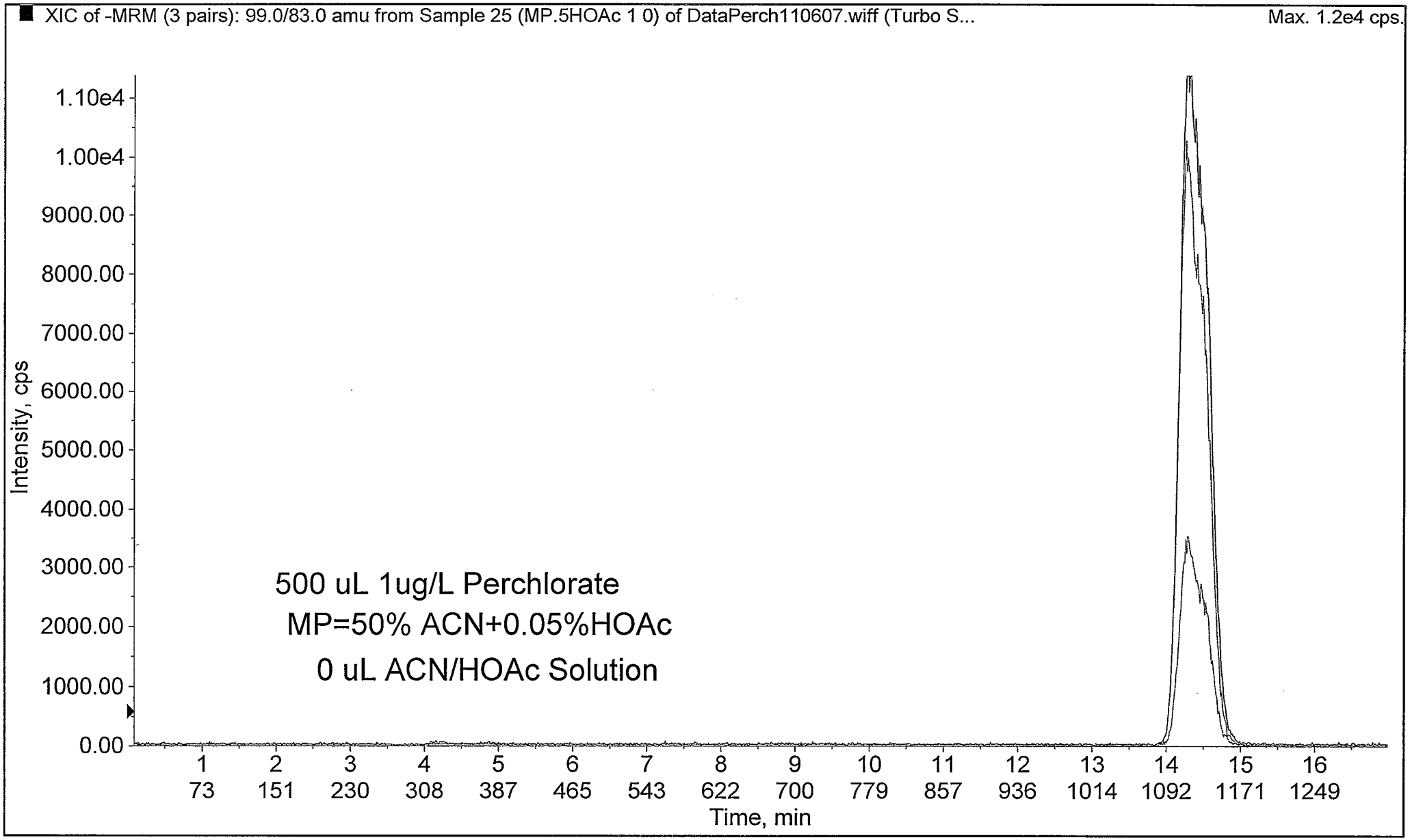

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 8 (.01 500+100) of DataPerch110607.wiff (Turbo Spra...
Max. 196.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL 0.01ug/L Perchlorate
MP=50% ACN+0.1%HOAc
100 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

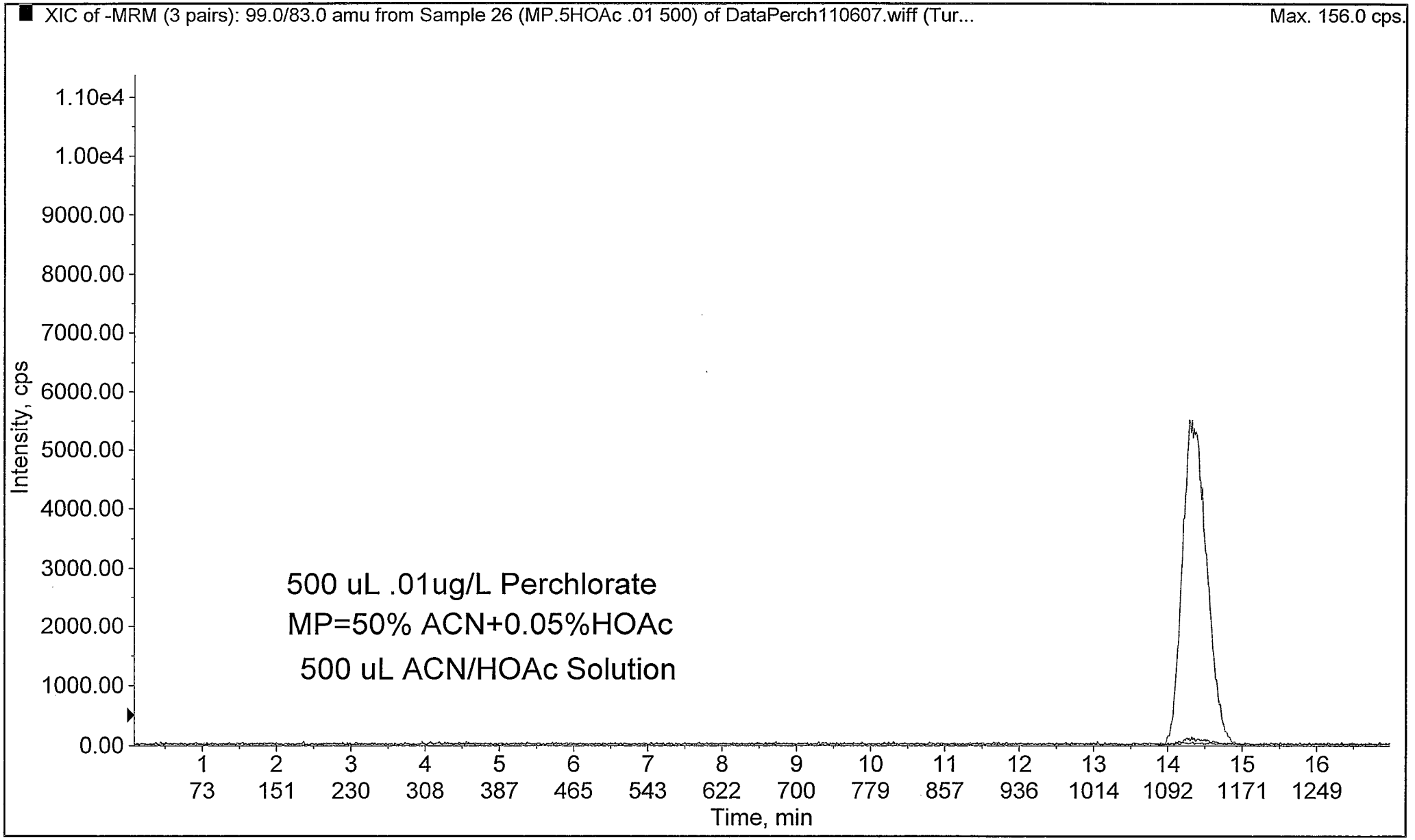

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 9 (.01 500+0) of DataPerch110607.wiff (Turbo Spray)
Max. 196.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL 0.01ug/L Perchlorate
MP=50% ACN+0.1%HOAc
0 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

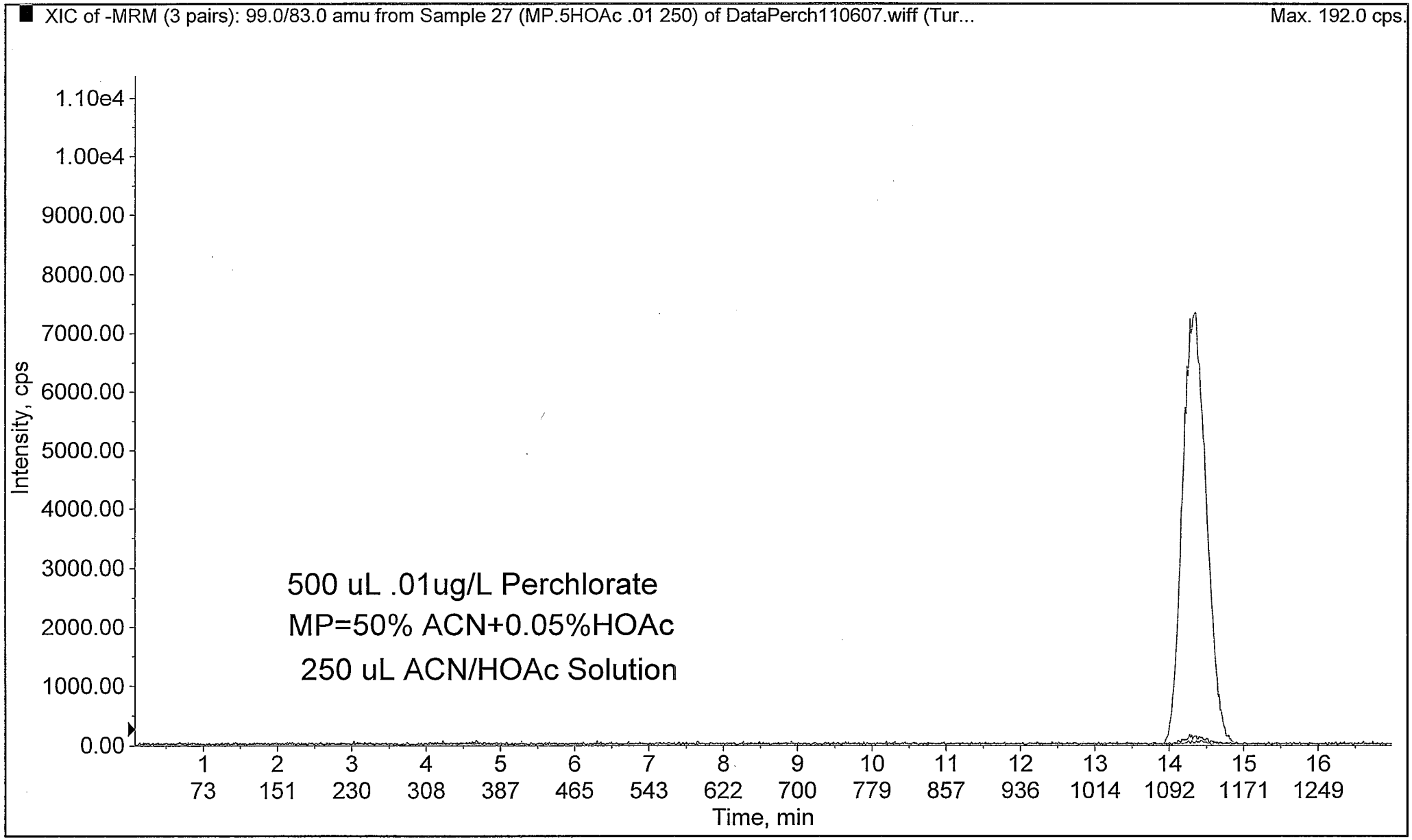

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 22 (MP.5HOAc 1 500) of DataPerch110607.wiff (Tur...
Max. 6088.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
14.19
500 uL 1ug/L Perchlorate
MP=50% ACN+0.05%HOAc
500 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

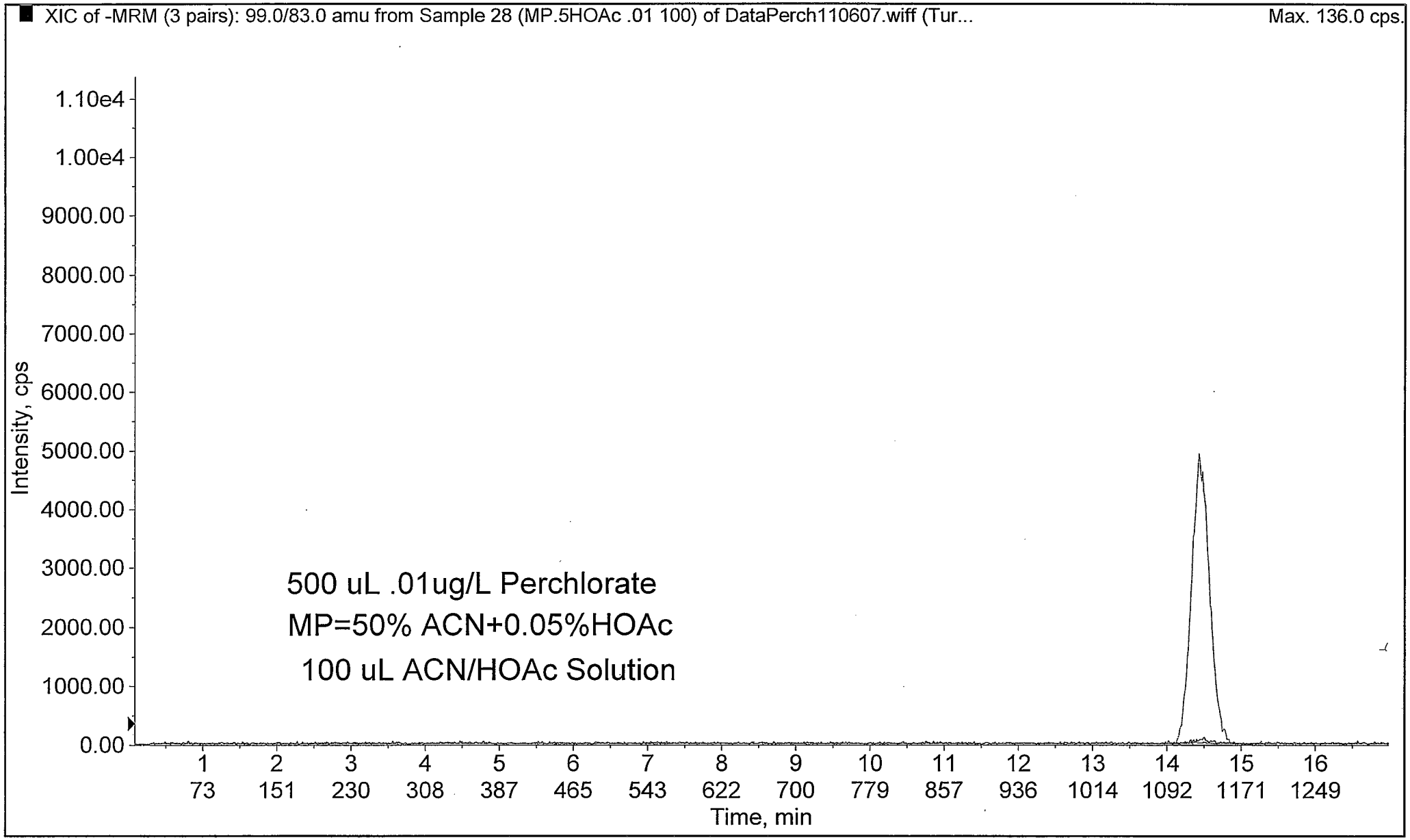

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 23 (MP.5HOAc 1 250) of DataPerch110607.wiff (Tur...
Max. 8636.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
14.32
500 uL 1ug/L Perchlorate
MP=50% ACN+0.05%HOAc
250 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 24 (MP.5HOAc 1 100) of DataPerch110607.wiff (Turb...
Max. 1.1e4 cps.
14.36
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
Intensity, cps
500 uL 1ug/L Perchlorate
MP=50% ACN+0.05%HOAc
100 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 25 (MP.5HOAc 1 0) of DataPerch110607.wiff (Turbo S...
Max. 1.2e4 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL 1ug/L Perchlorate
MP=50% ACN+0.05%HOAc
0 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 26 (MP.5HOAc .01 500) of DataPerch110607.wiff (Tur...
Max. 156.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL .01ug/L Perchlorate
MP=50% ACN+0.05%HOAc
500 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

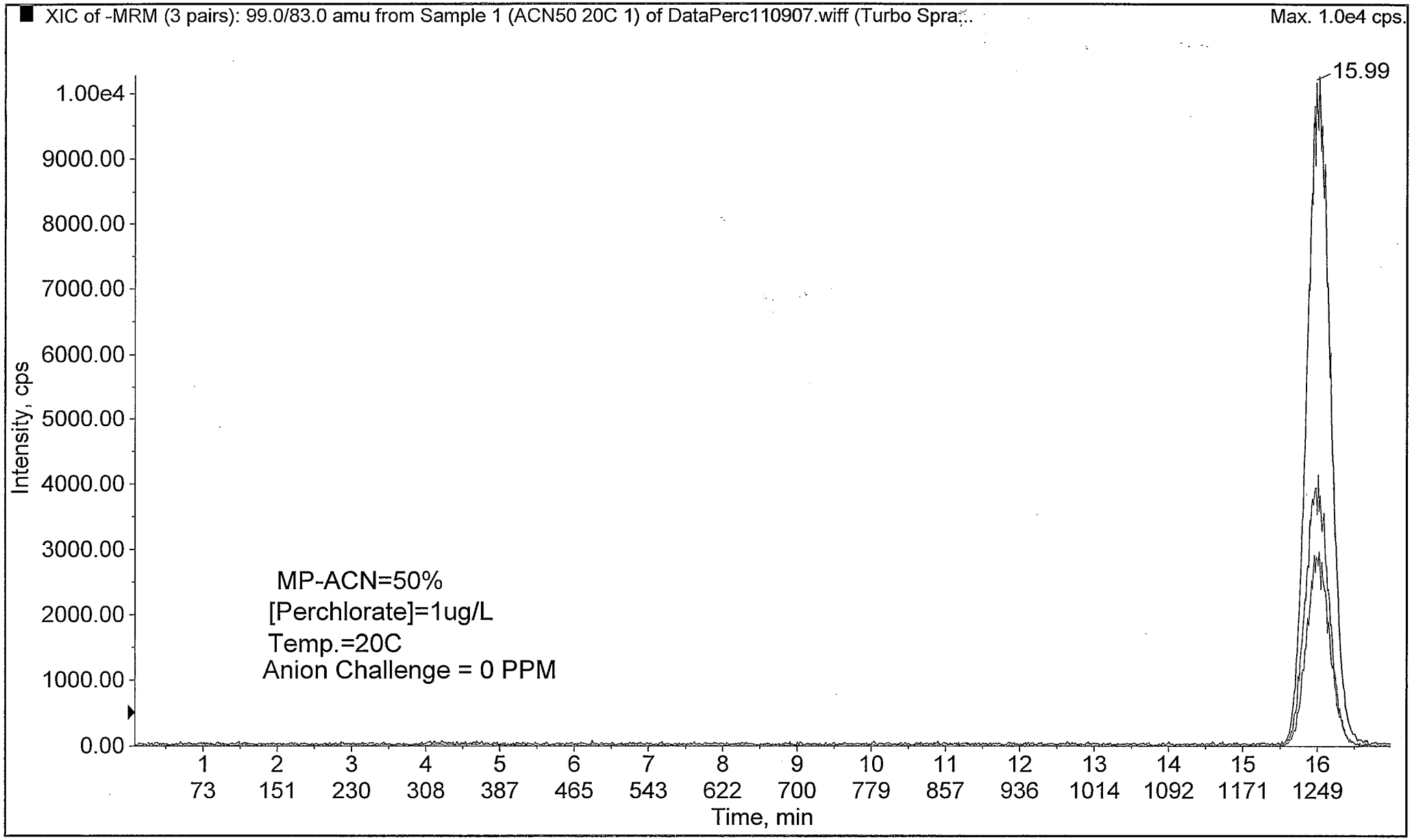

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 27 (MP.5HOAc .01 250) of DataPerch110607.wiff (Tur...
Max. 192.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL .01ug/L Perchlorate
MP=50% ACN+0.05%HOAc
250 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

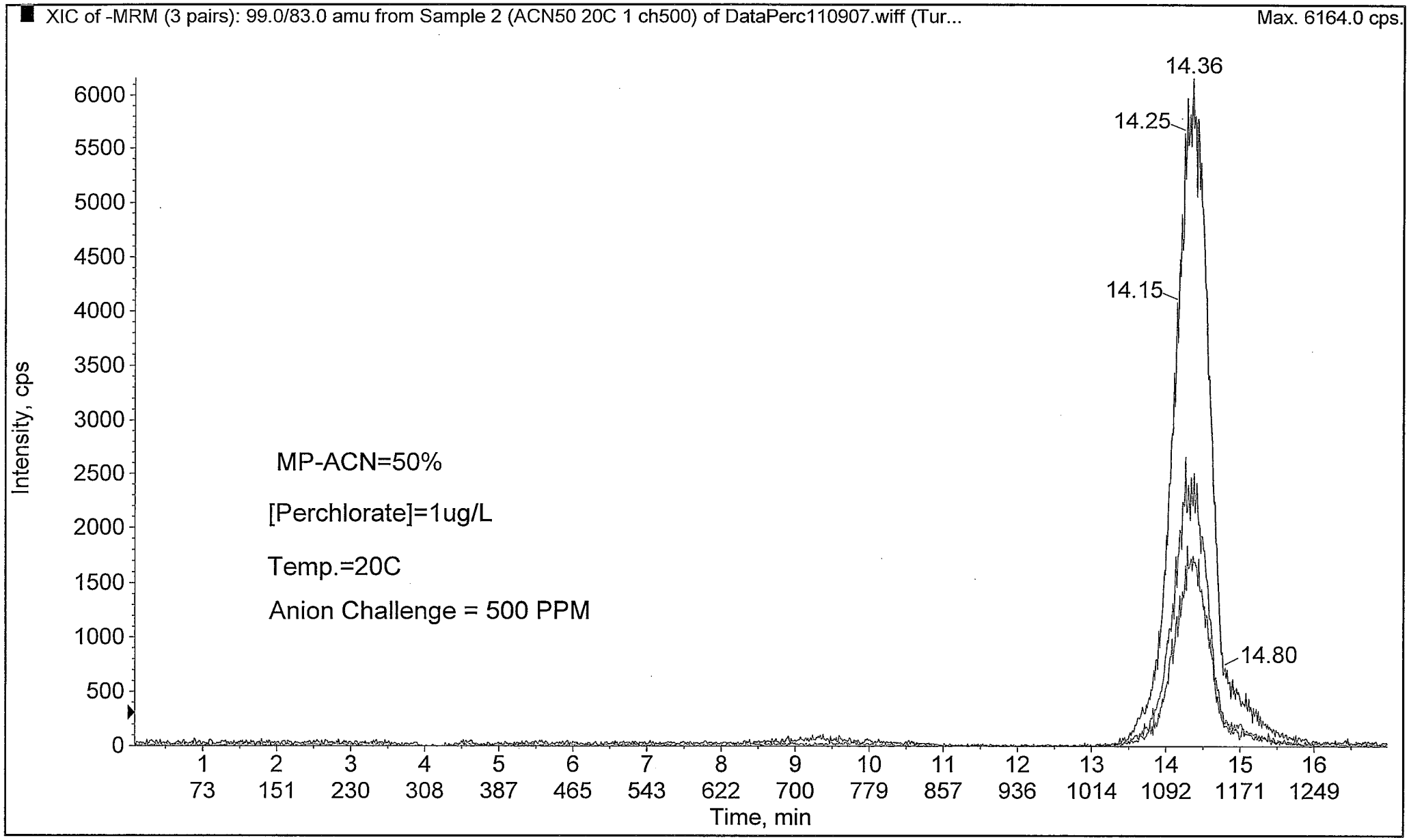

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 28 (MP.5HOAc .01 100) of DataPerch110607.wiff (Tur...
Max. 136.0 cps.
Intensity, cps
1.10e4
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
500 uL .01ug/L Perchlorate
MP=50% ACN+0.05%HOAc
100 uL ACN/HOAc Solution
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

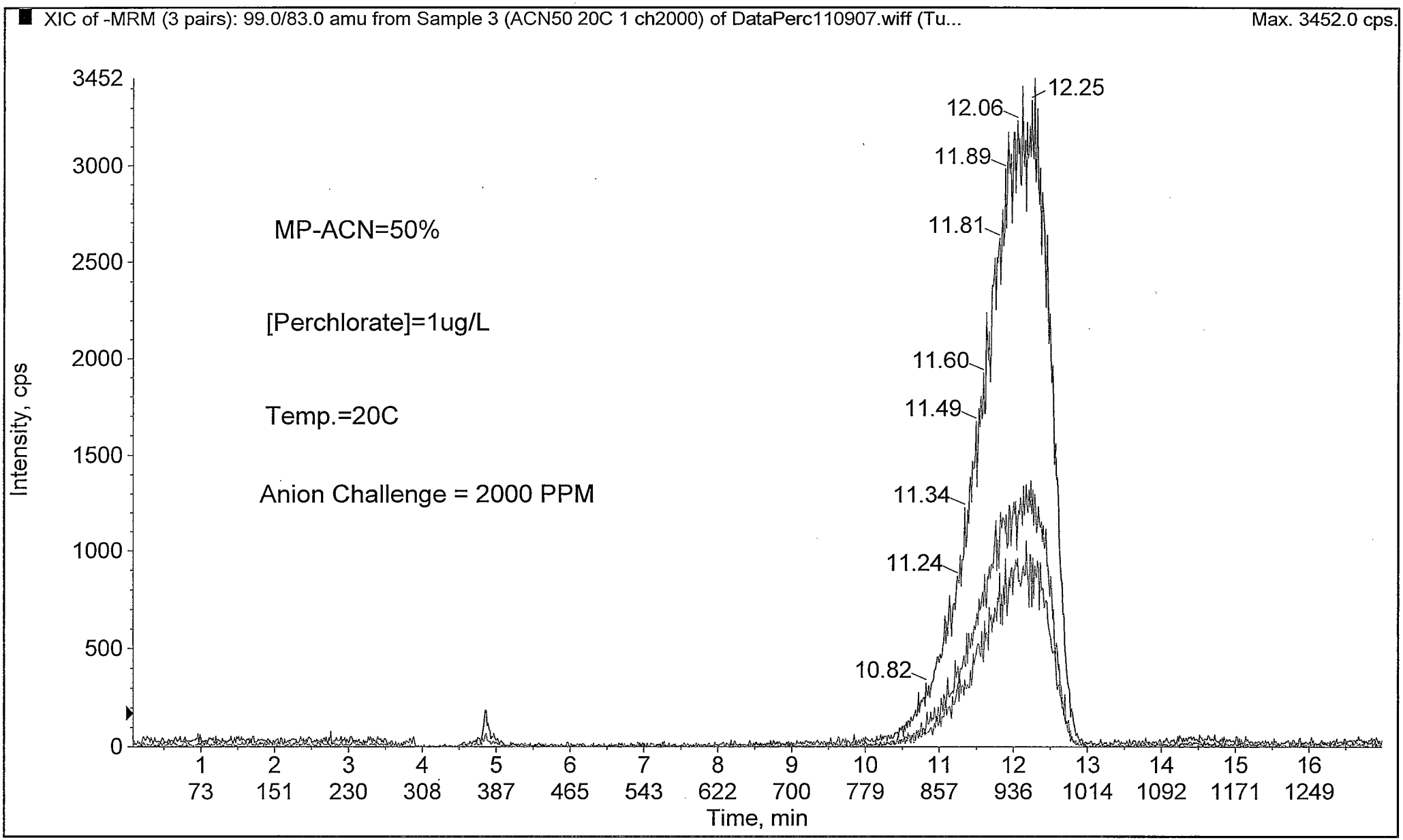
Appendix IV

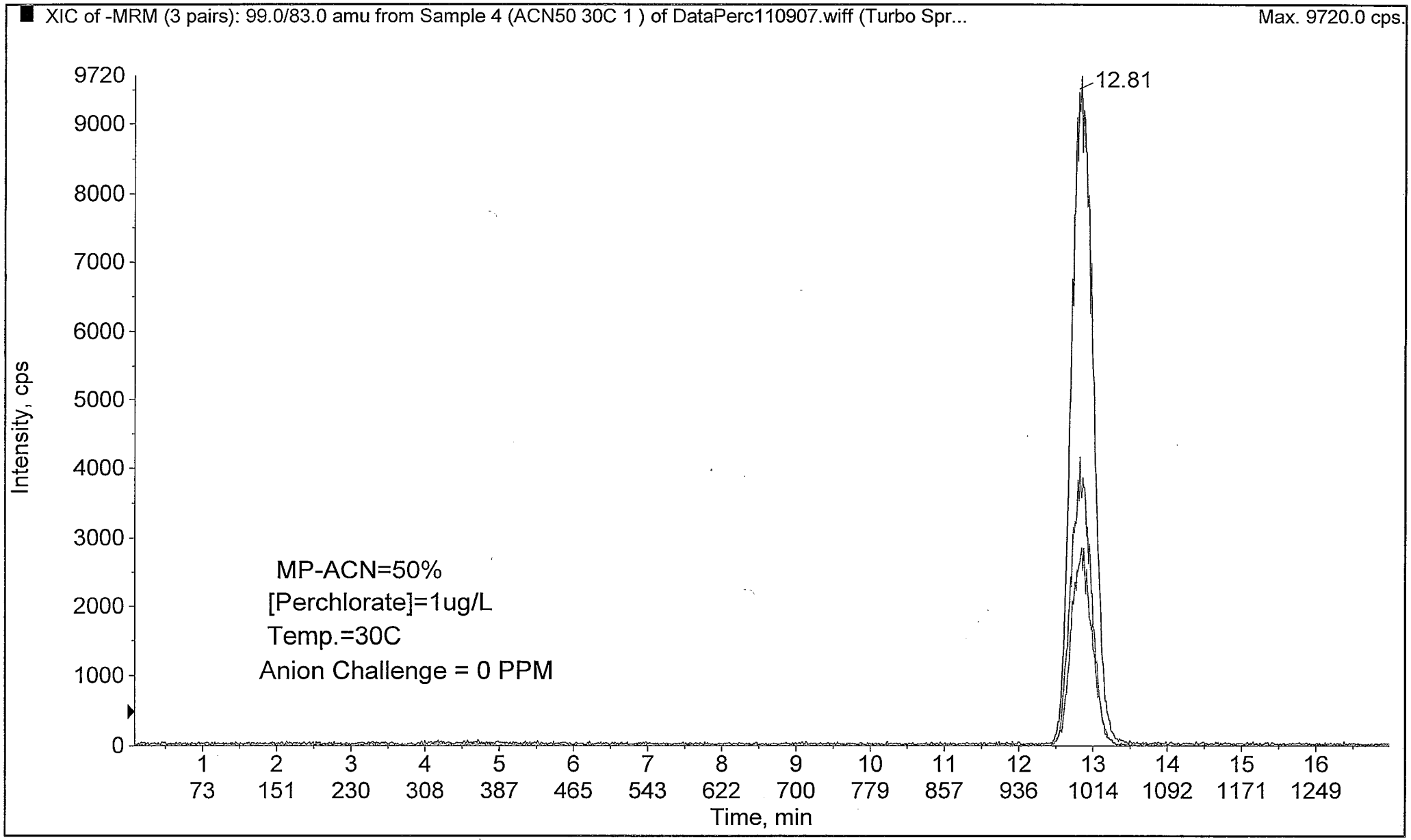
Effect of Temperature and Anion Challenge

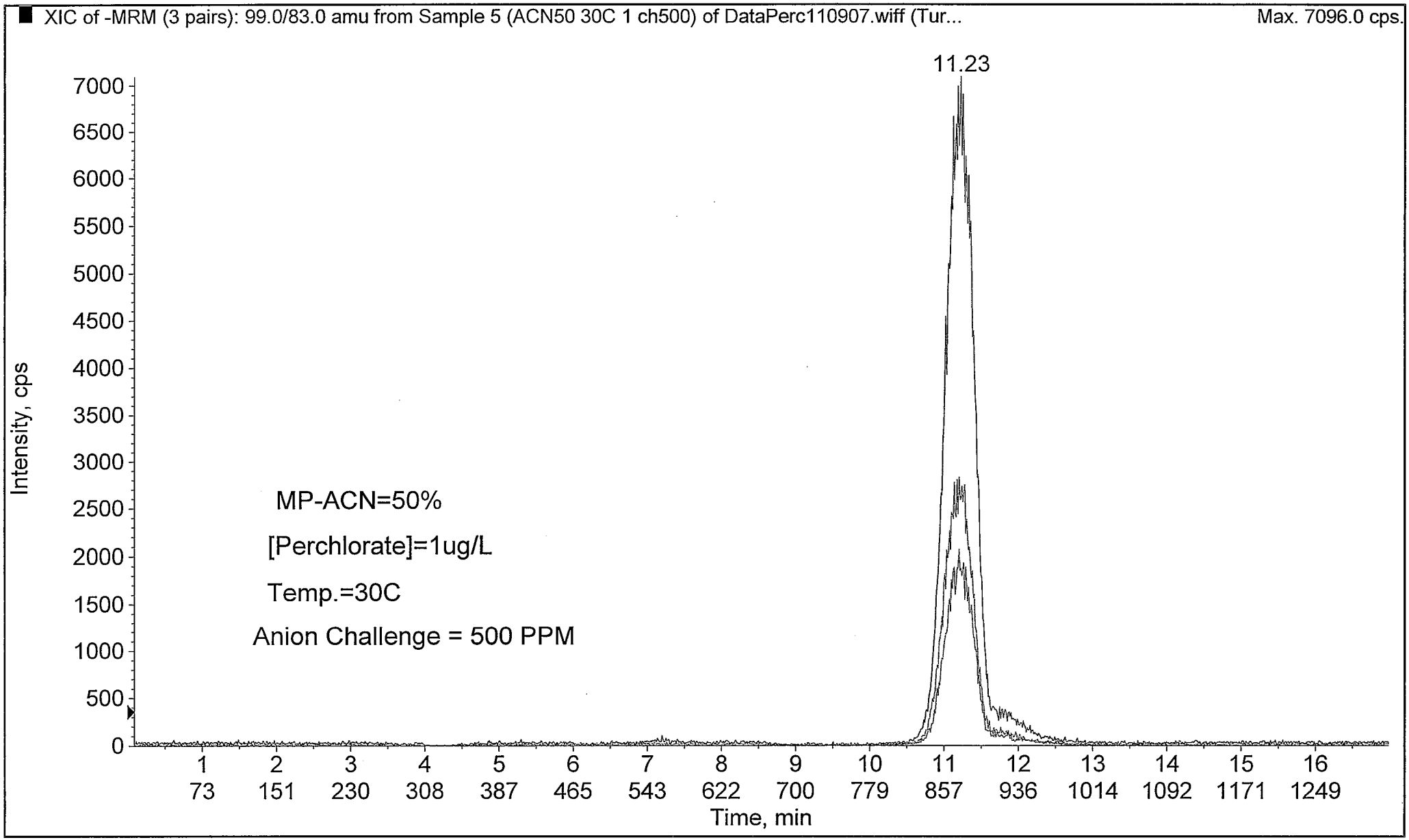
Effect of Temperature &
Anion Challenge

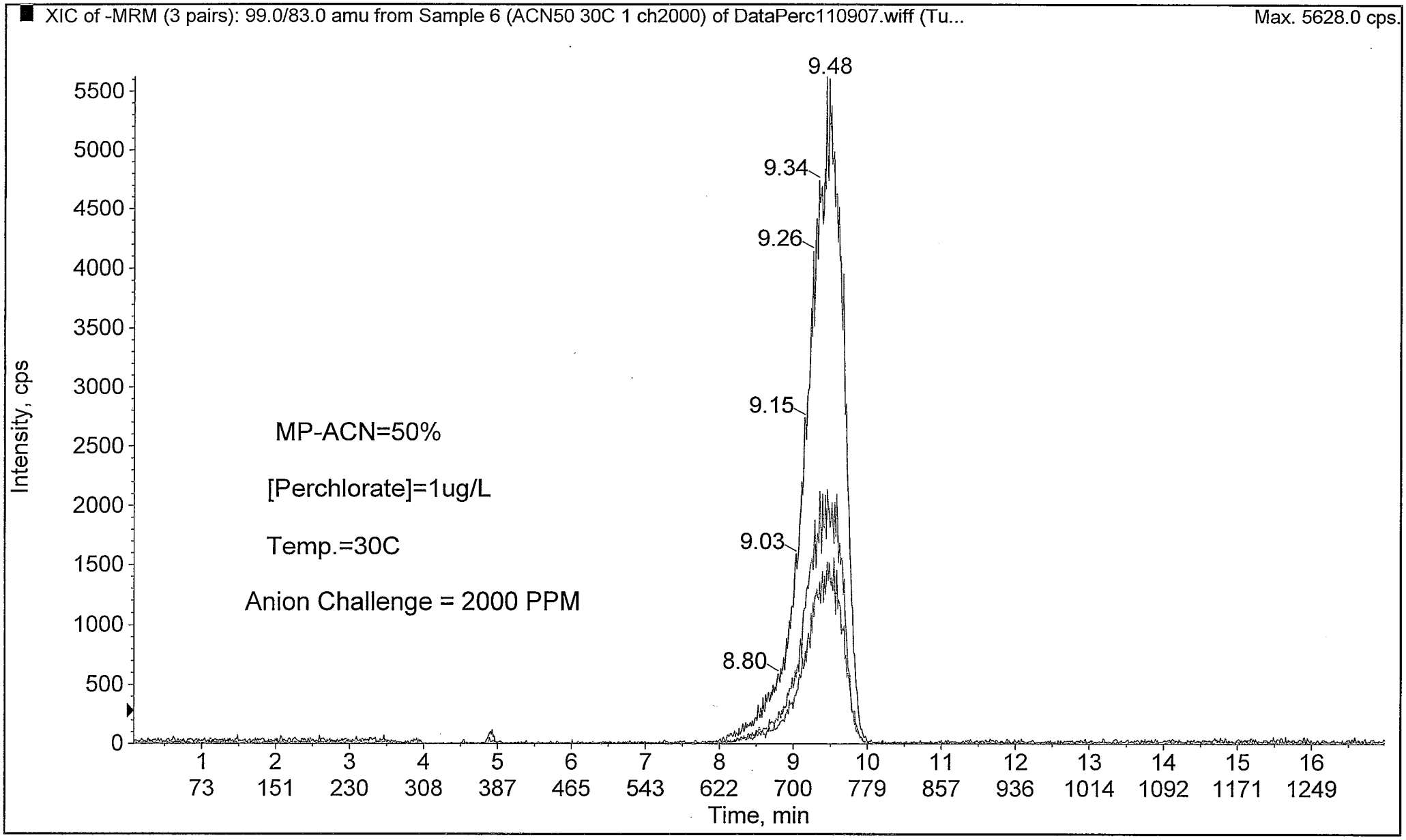

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 1 (ACN50 20C 1) of DataPerc110907.wiff (Turbo Spra...
Max. 1.0e4 cps.
15.99
1.00e4
9000.00
8000.00
7000.00
6000.00
5000.00
4000.00
3000.00
2000.00
1000.00
0.00
Intensity, cps
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=20C
Anion Challenge = 0 PPM
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

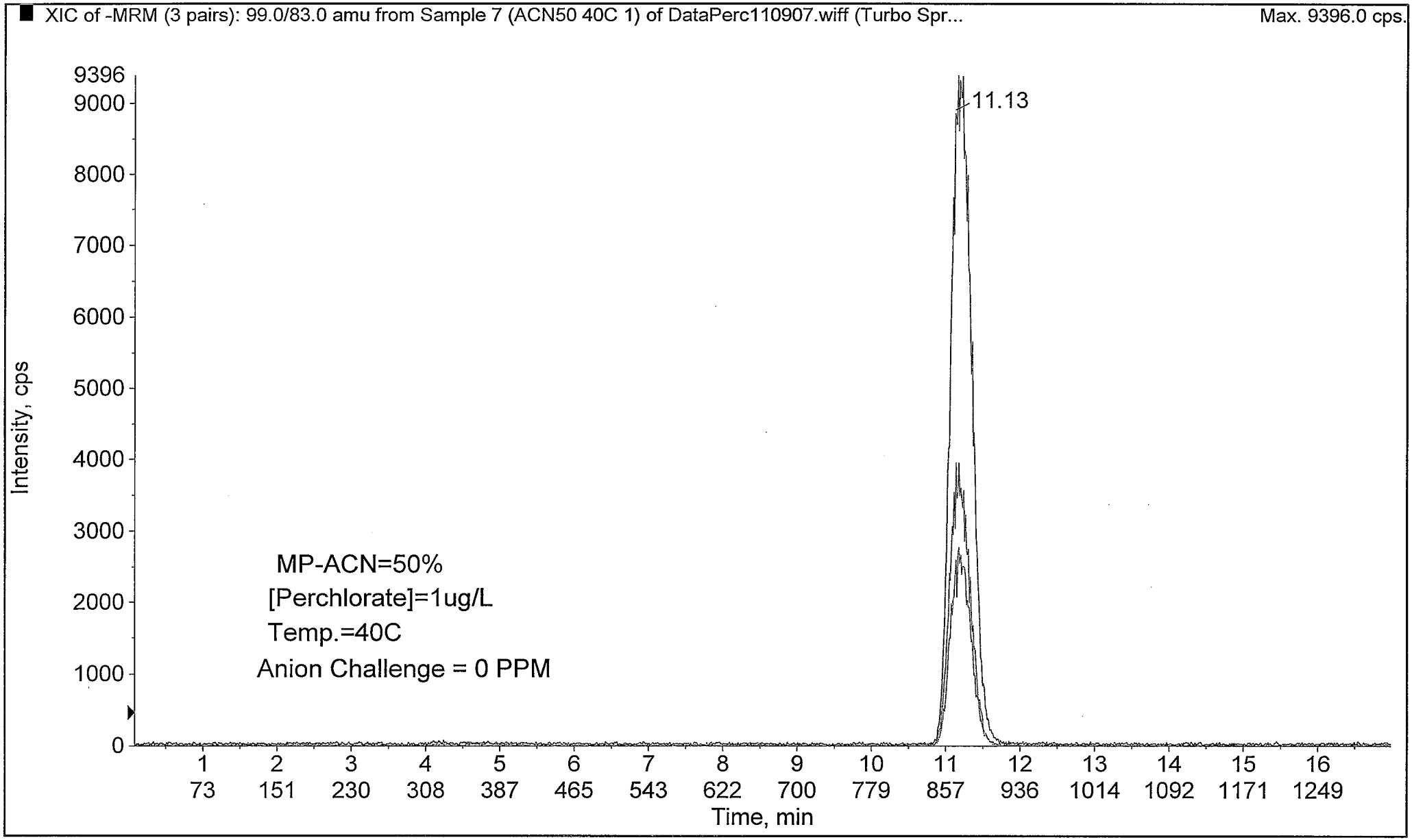

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 2 (ACN50 20C 1 ch500) of DataPerc110907.wiff (Tur...
Max. 6164.0 cps.
14.36
14.25
14.15
14.80
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=20C
Anion Challenge = 500 PPM
Intensity, cps
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

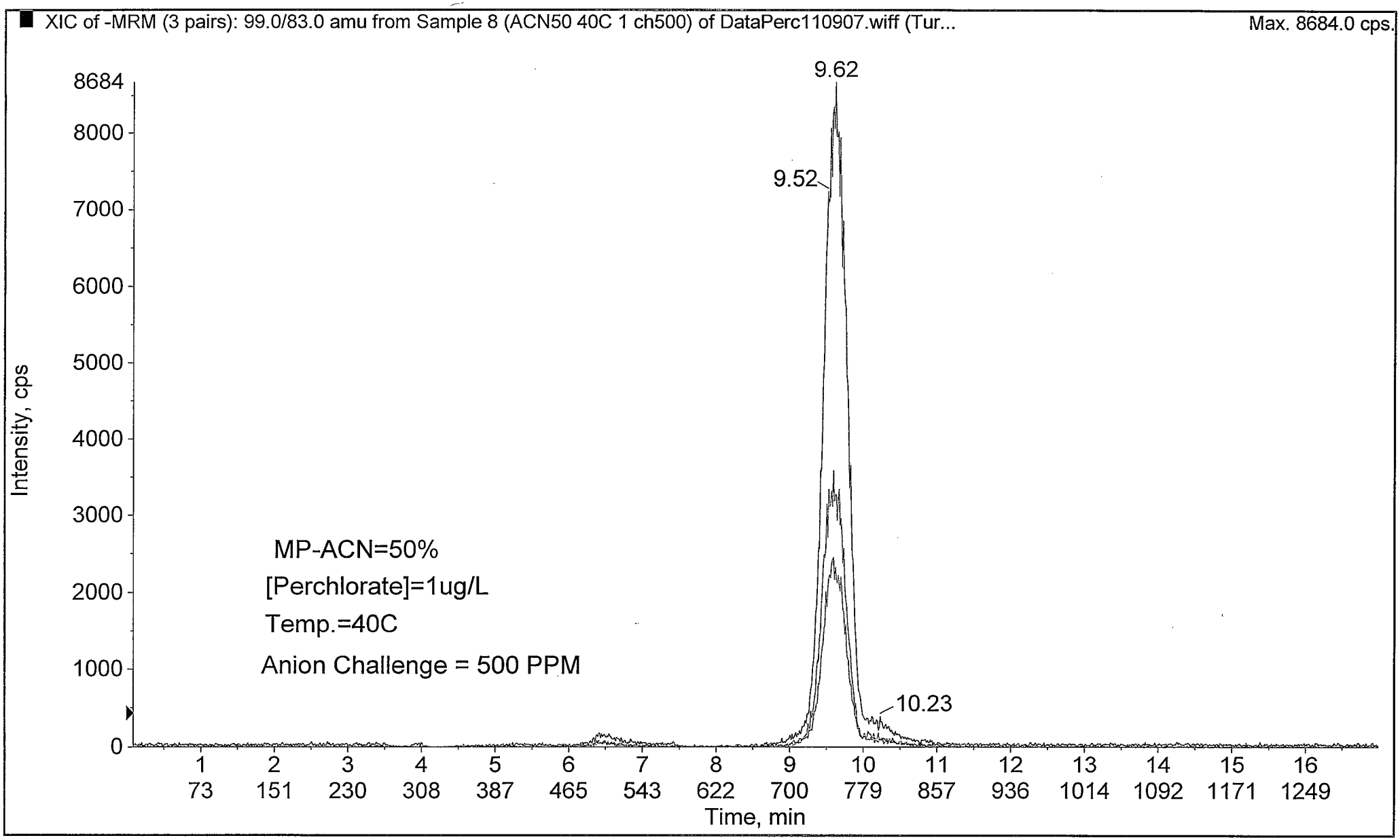

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 3 (ACN50 20C 1 ch2000) of DataPerc110907.wiff (Tu...
Max. 3452.0 cps.
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=20C
Anion Challenge = 2000 PPM
12.25
12.06
11.89
11.81
11.60
11.49
11.34
11.24
10.82
Intensity, cps
Time, min

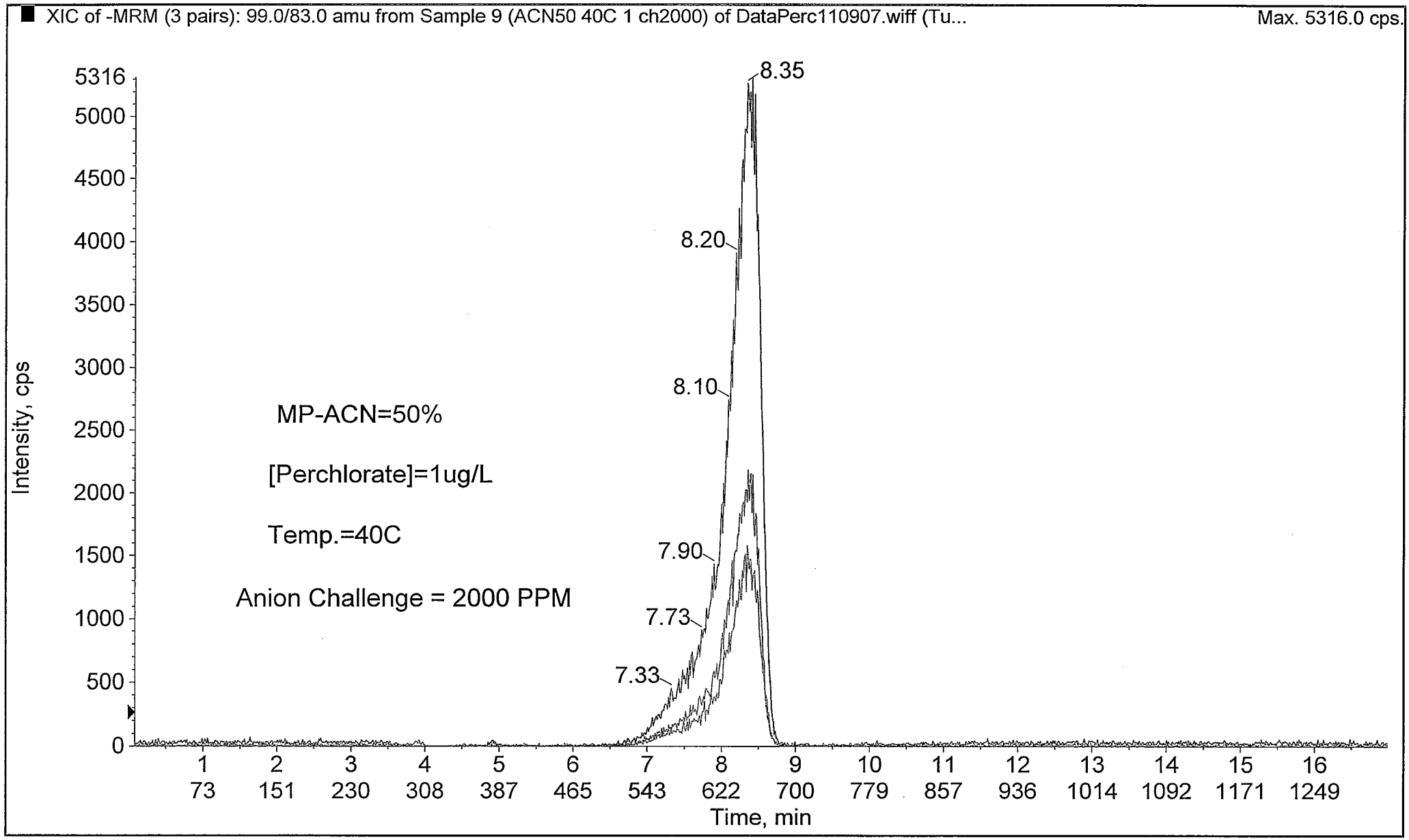

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 4 (ACN50 30C 1) of DataPerc110907.wiff (Turbo Spr...
Max. 9720.0 cps.
12.81
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=30C
Anion Challenge = 0 PPM
Intensity, cps
9720
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

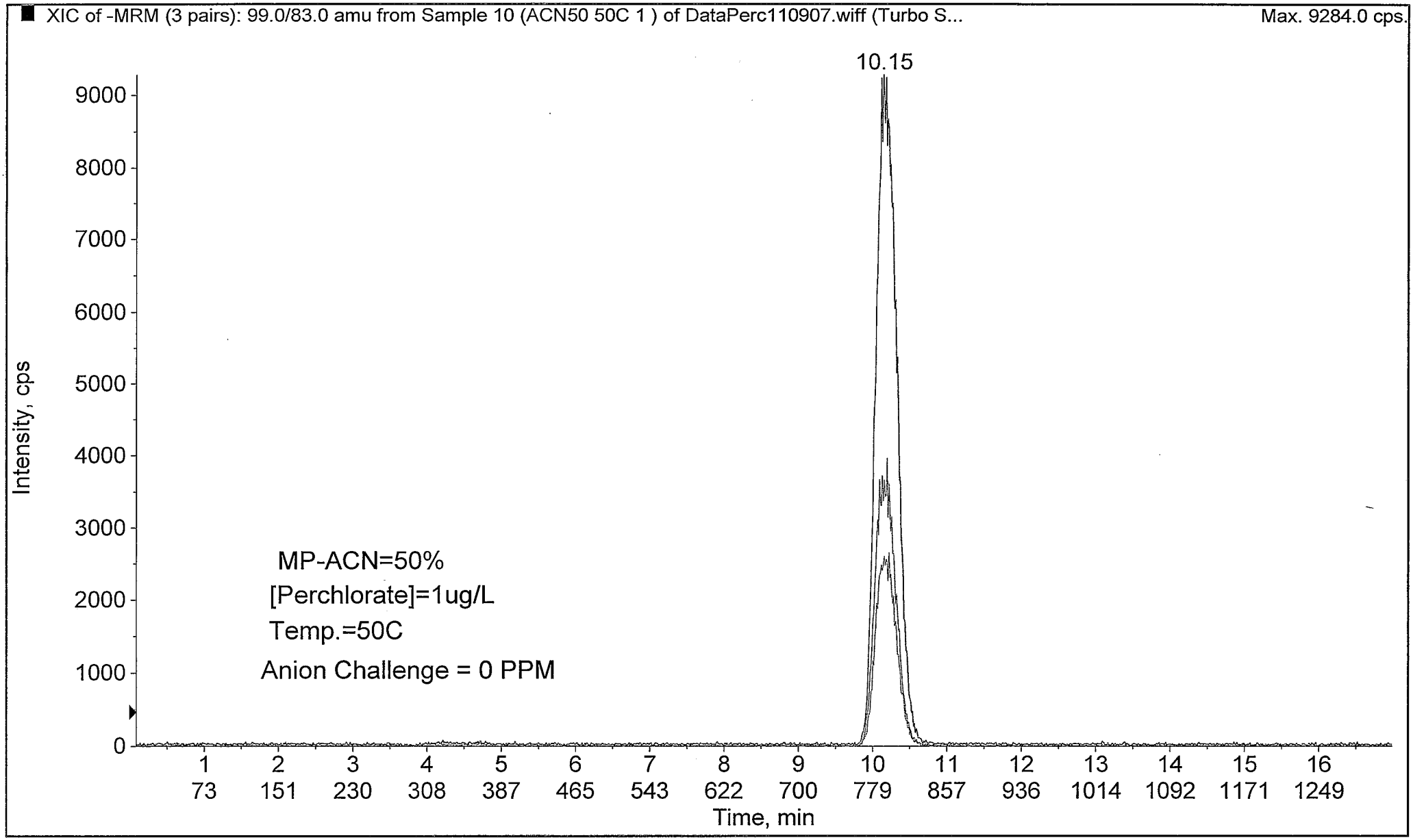

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 5 (ACN50 30C 1 ch500) of DataPerc110907.wiff (Tur...
Max. 7096.0 cps.
11.23
Intensity, cps
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=30C
Anion Challenge = 500 PPM
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

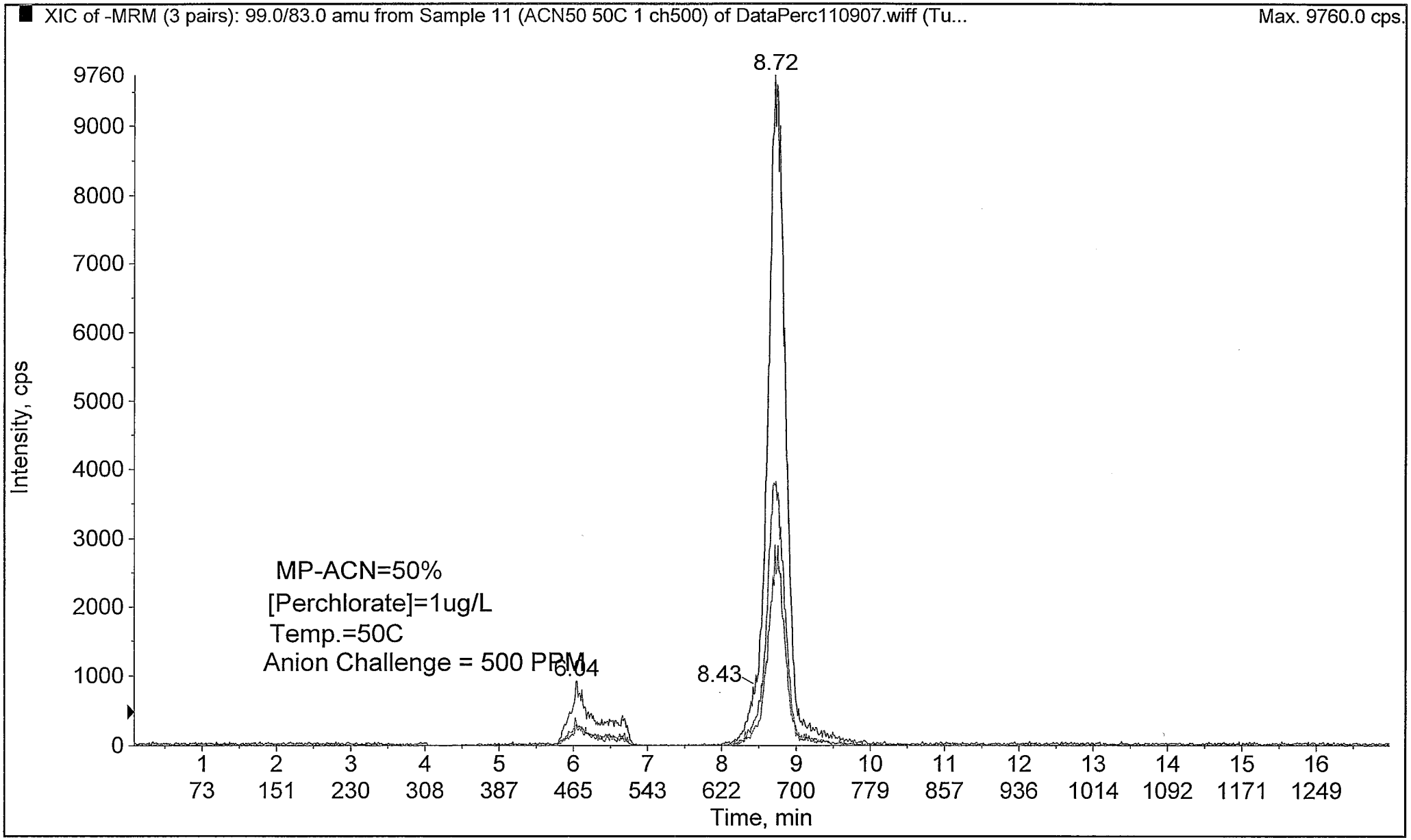

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 6 (ACN50 30C 1 ch2000) of DataPerc110907.wiff (Tu...
Max. 5628.0 cps.
9.48
9.34
9.26
9.15
9.03
8.80
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=30C
Anion Challenge = 2000 PPM
Intensity, cps
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

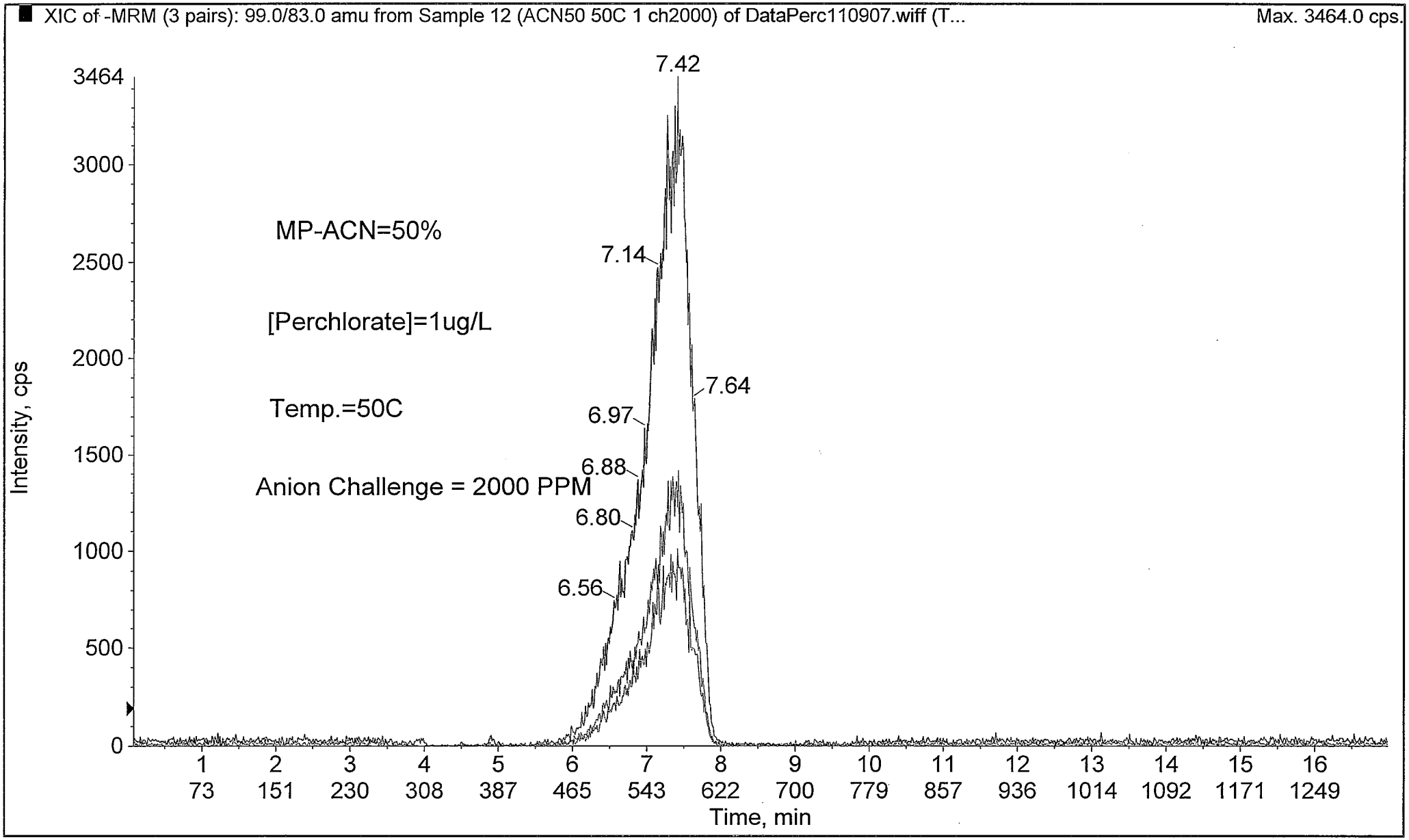

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 7 (ACN50 40C 1) of DataPerc110907.wiff (Turbo Spr...
Max. 9396.0 cps.
9396
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
Intensity, cps
11.13
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=40C
Anion Challenge = 0 PPM
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 8 (ACN50 40C 1 ch500) of DataPerc110907.wiff (Tur...
Max. 8684.0 cps.
9.62
9.52
10.23
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=40C
Anion Challenge = 500 PPM
Intensity, cps
8684
8000
7000
6000
5000
4000
3000
2000
1000
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 9 (ACN50 40C 1 ch2000) of DataPerc110907.wiff (Tu...
Max. 5316.0 cps.
Intensity, cps
5316
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
8.35
8.20
8.10
7.90
7.73
7.33
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=40C
Anion Challenge = 2000 PPM
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

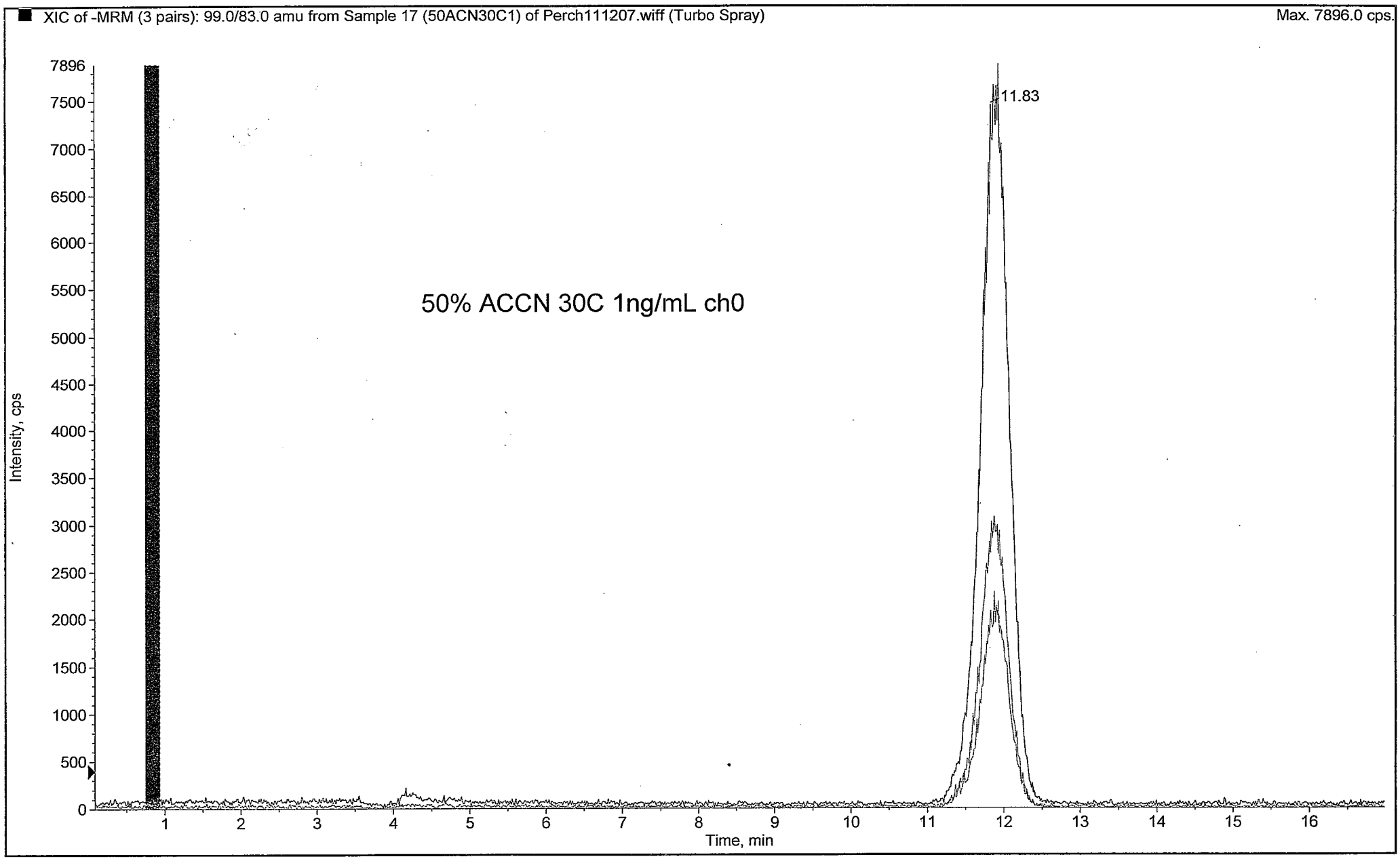

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 10 (ACN50 50C 1) of DataPerc110907.wiff (Turbo S...
Max. 9284.0 cps.
10.15
Intensity, cps
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=50C
Anion Challenge = 0 PPM
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min


XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 11 (ACN50 50C 1 ch500) of DataPerc110907.wiff (Tu...
Max. 9760.0 cps.
8.72
9760
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
Intensity, cps
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=50C
Anion Challenge = 500 PPM
6.04
8.43
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

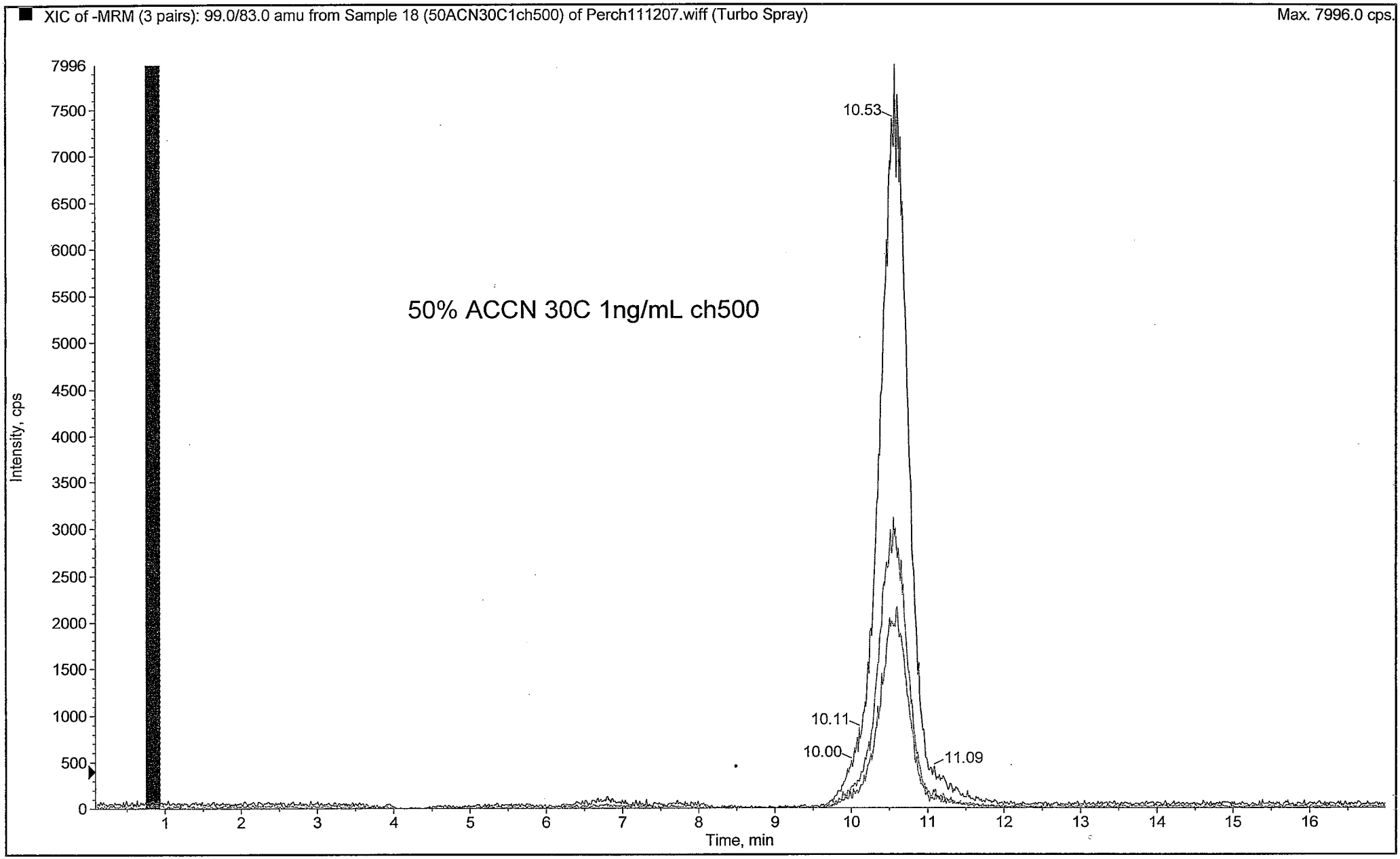

XIC of -MRM (3 pairs): 99.0/83.0 amu from Sample 12 (ACN50 50C 1 ch2000) of DataPerc110907.wiff (T...
Max. 3464.0 cps.
Intensity, cps
3464
3000
2500
2000
1500
1000
500
0
MP-ACN=50%
[Perchlorate]=1ug/L
Temp.=50C
Anion Challenge = 2000 PPM
7.42
7.14
7.64
6.97
6.88
6.80
6.56
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16
73 151 230 308 387 465 543 622 700 779 857 936 1014 1092 1171 1249
Time, min

MP 50/50
Temp 30
Challenge 0-2000

Operator: jefferie
Printing Time: 8:15:19 AM
Printing Date: Tuesday, November 13, 2007



Operator: jefferie
Printing Time: 8:15:38 AM
Printing Date: Tuesday, November 13, 2007



Operator: jefferie
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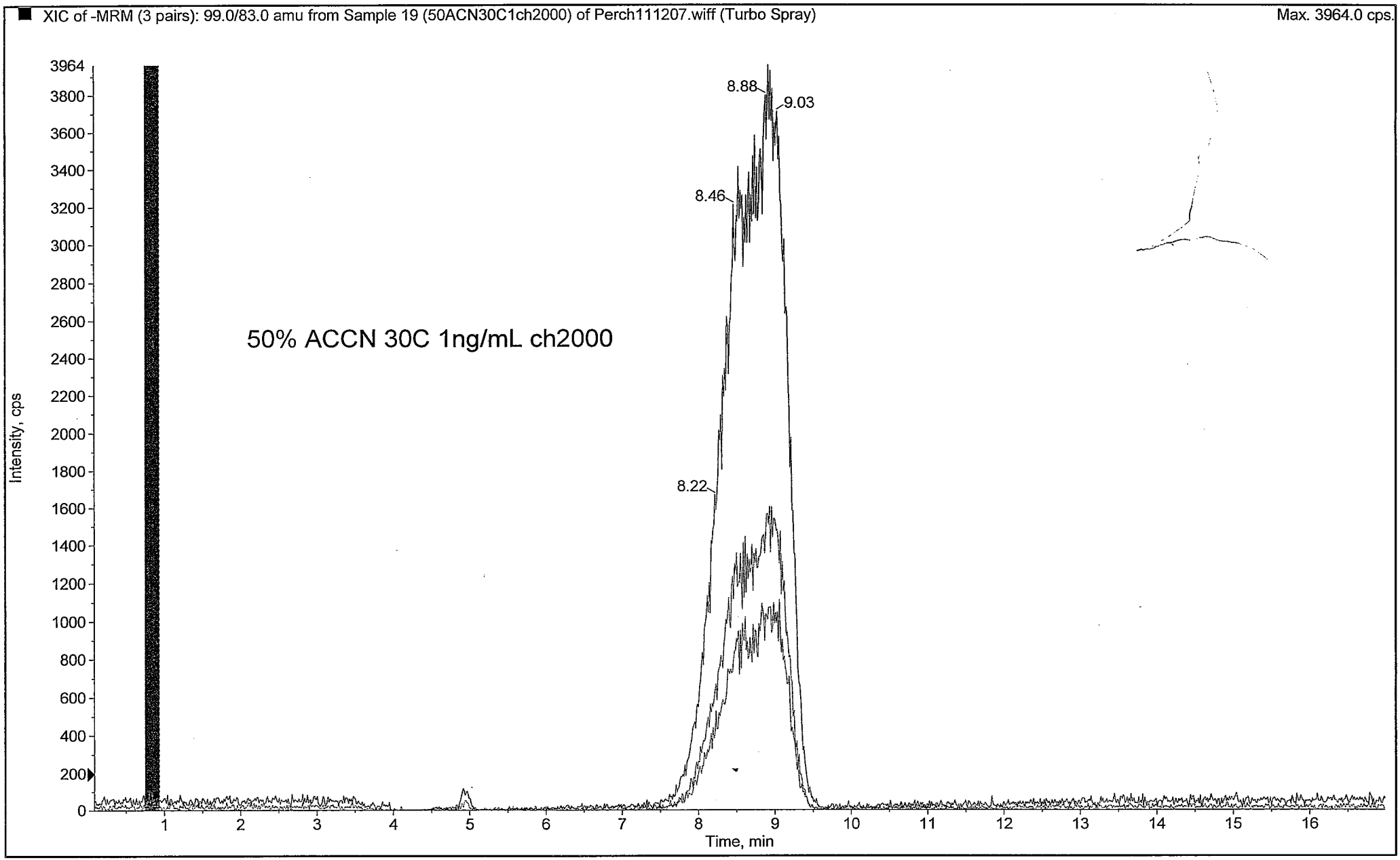
Printing Date: Tuesday, November 13, 2007



Isotope Ratios for 11/28/2007 ICAL VS ICS								
	Sample Na	Perc 1	Perc 2	Isotope Ratio				
	S0.01	6.05E+03	1.75E+03	3.45				
	S0.05	1.45E+04	4.49E+03	3.22				
	S0.1	3.13E+04	9.56E+03	3.27				
	S1.0	3.04E+05	9.18E+04	3.31				
	S10	2.59E+06	7.90E+05	3.28				
	S50	1.10E+07	3.43E+06	3.21				
	S100	1.94E+07	6.12E+06	3.18				
			AVE ICAL	3.27				
	ITS500	2.29E+05	6.73E+04	3.40				
	ITS2000	2.67E+05	8.32E+04	3.21				
	ITS4000	3.08E+05	9.73E+04	3.16				
	ITS6000	6.20E+03	1.98E+03	3.14				
	ITS500	2.64E+05	7.81E+04	3.38				
	ITS4000	3.15E+05	1.00E+05	3.14				
	ITS4500	2.23E+05	6.81E+04	3.28				
	ITS5000	6.45E+04	1.94E+04	3.32				
	ITS5500	1.03E+04	2.88E+03	3.56				
	ITS6000	6.18E+03	1.77E+03	3.50				
			AVE ICS	3.31				

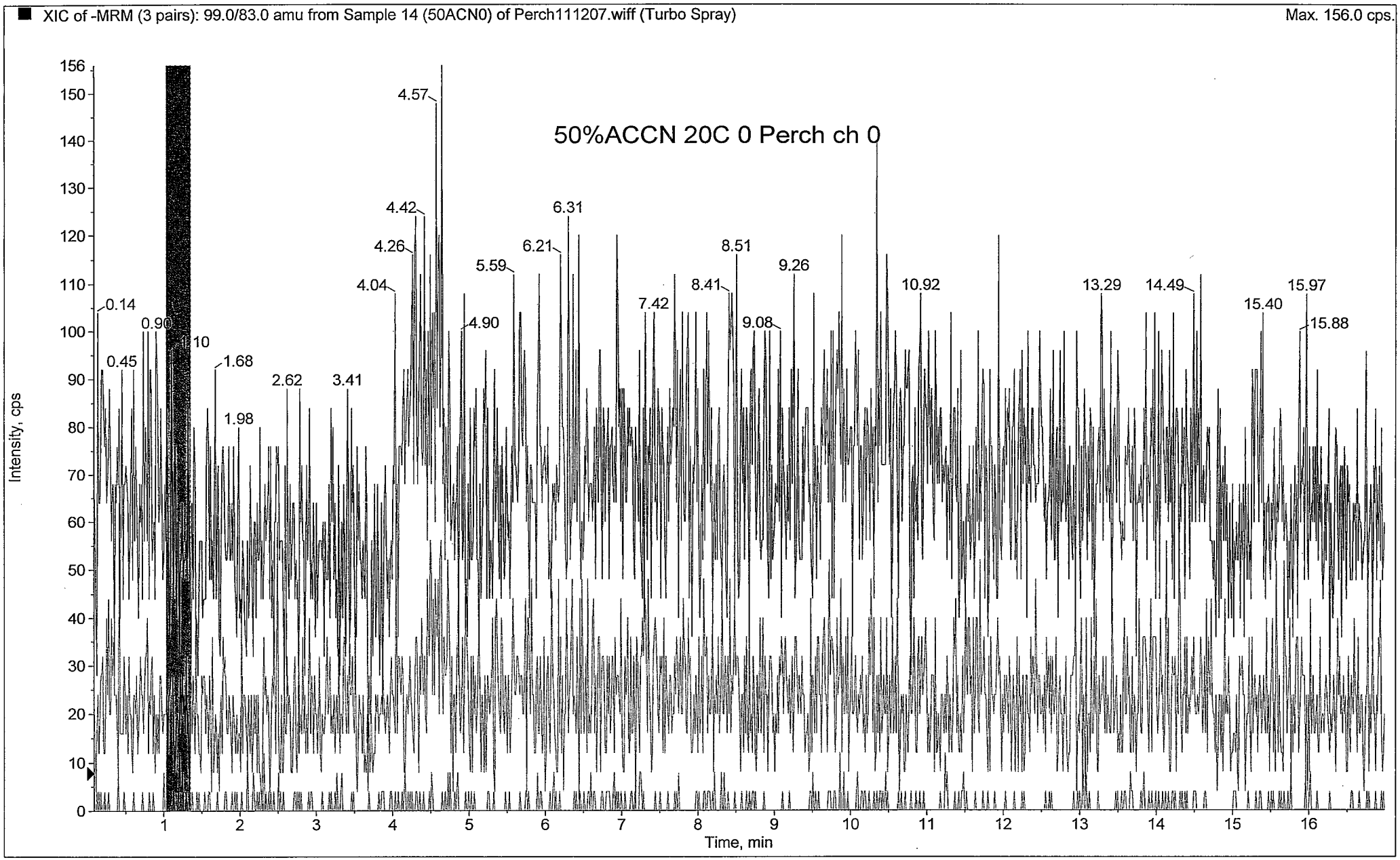
Isotope Ratios for 121107

	Sample Na	Perc 1	Perc 2	Isotope Ratio		
1	CCB	0.00E+00	0.00E+00			
2	S0.01	2.96E+03	9.50E+02	3.11		
3	S0.05	7.85E+03	2.59E+03	3.04		
4	S0.1	1.71E+04	5.45E+03	3.14		
5	S0.5	8.02E+04	2.43E+04	3.29		
6	S1.0	1.72E+05	5.25E+04	3.27		
7	S5.0	7.55E+05	2.32E+05	3.25		
8	S10	1.48E+06	4.52E+05	3.27		
9	S50	6.97E+06	2.16E+06	3.23		
10	S100	1.31E+07	4.14E+06	3.16	Ave ICAL	3.20
11	ICV	1.66E+05	5.05E+04	3.28		
12	LODV-1	3.41E+03	1.16E+03	2.94		
13	LM071211-	0.00E+00	0.00E+00			
14	0711043-1	1.36E+06	4.25E+05	3.2		
15	0711043-1	1.31E+06	4.04E+05	3.25		
16	0711043-1	1.34E+06	4.15E+05	3.24		
17	CCV	1.54E+05	4.75E+04	3.25		
18	LODV-2	3.76E+03	1.19E+03	3.16		
19	CCV-2	1.11E+05	3.39E+04	3.29		
20	LM071211.	9.49E+03	3.06E+03	3.1		
21	ICS	8.79E+03	2.90E+03	3.03		

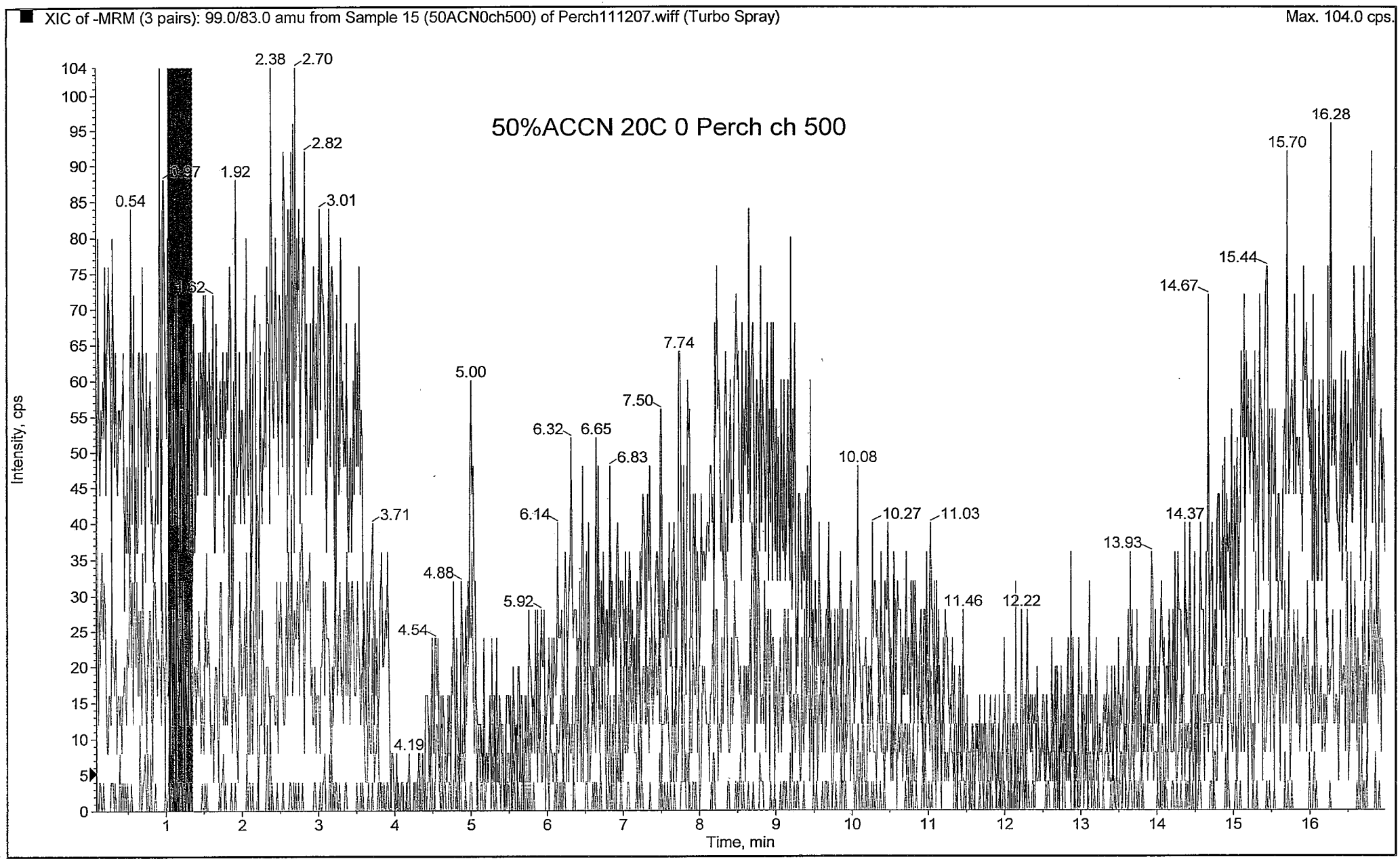
Appendix V

Effect of Anion Challenge on Baseline Noise

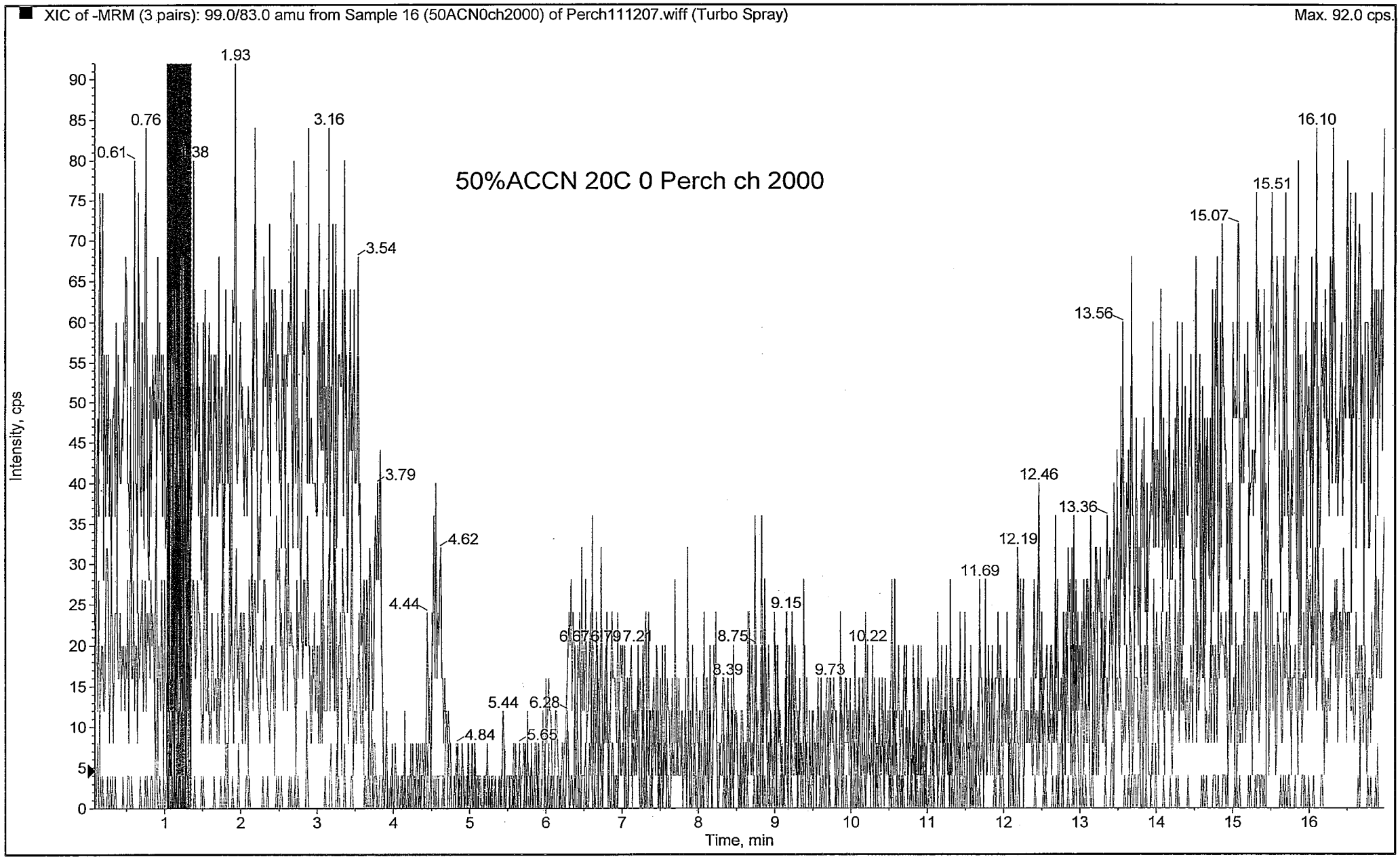
Operator: jefferie
Printing Time: 4:09:11 PM
Printing Date: Monday, November 12, 2007



Operator: jefferie
Printing Time: 4:09:42 PM
Printing Date: Monday, November 12, 2007



Operator: jefferie
Printing Time: 4:10:25 PM
Printing Date: Monday, November 12, 2007



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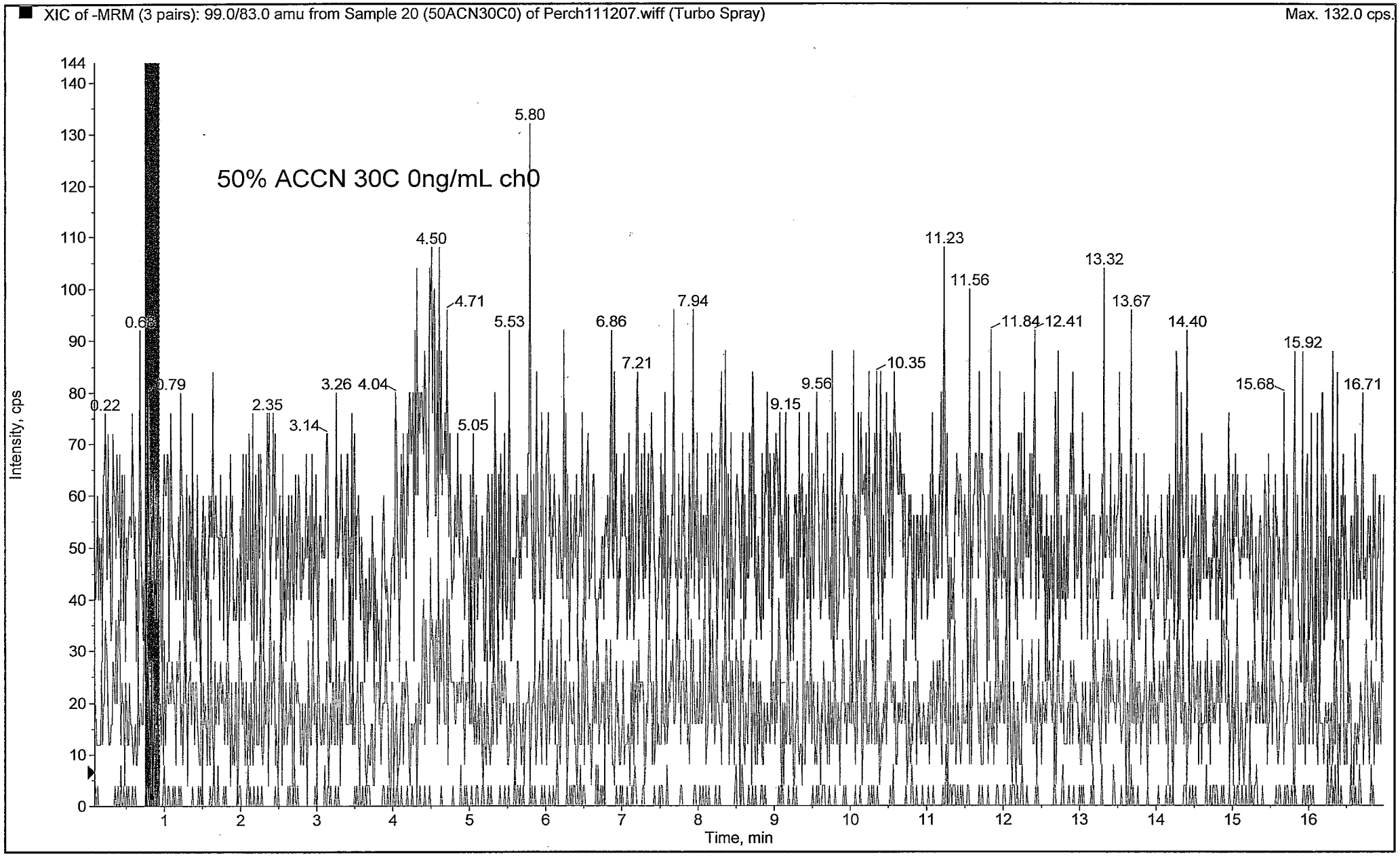
Printing Date: Tuesday, November 13, 2007



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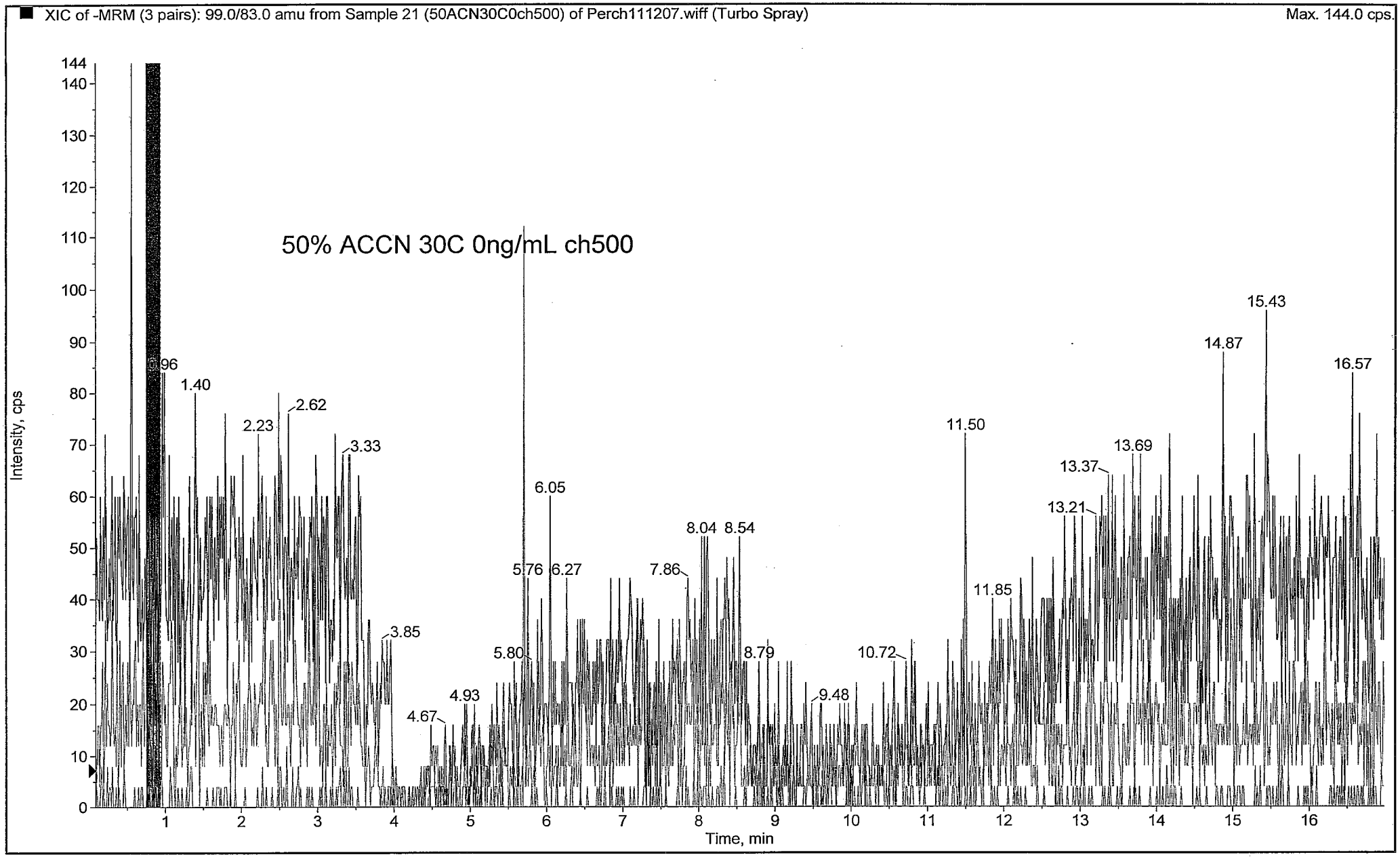

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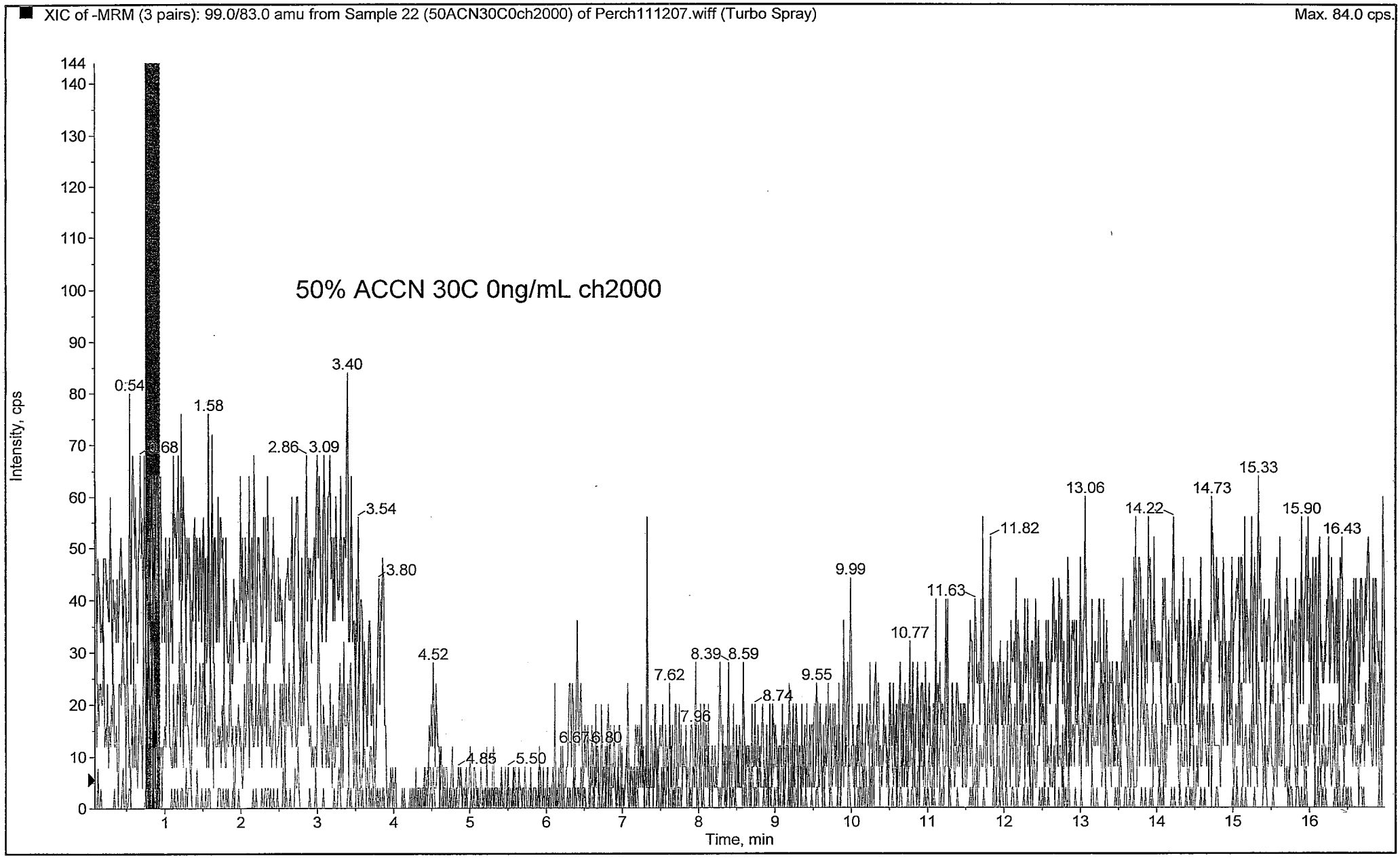

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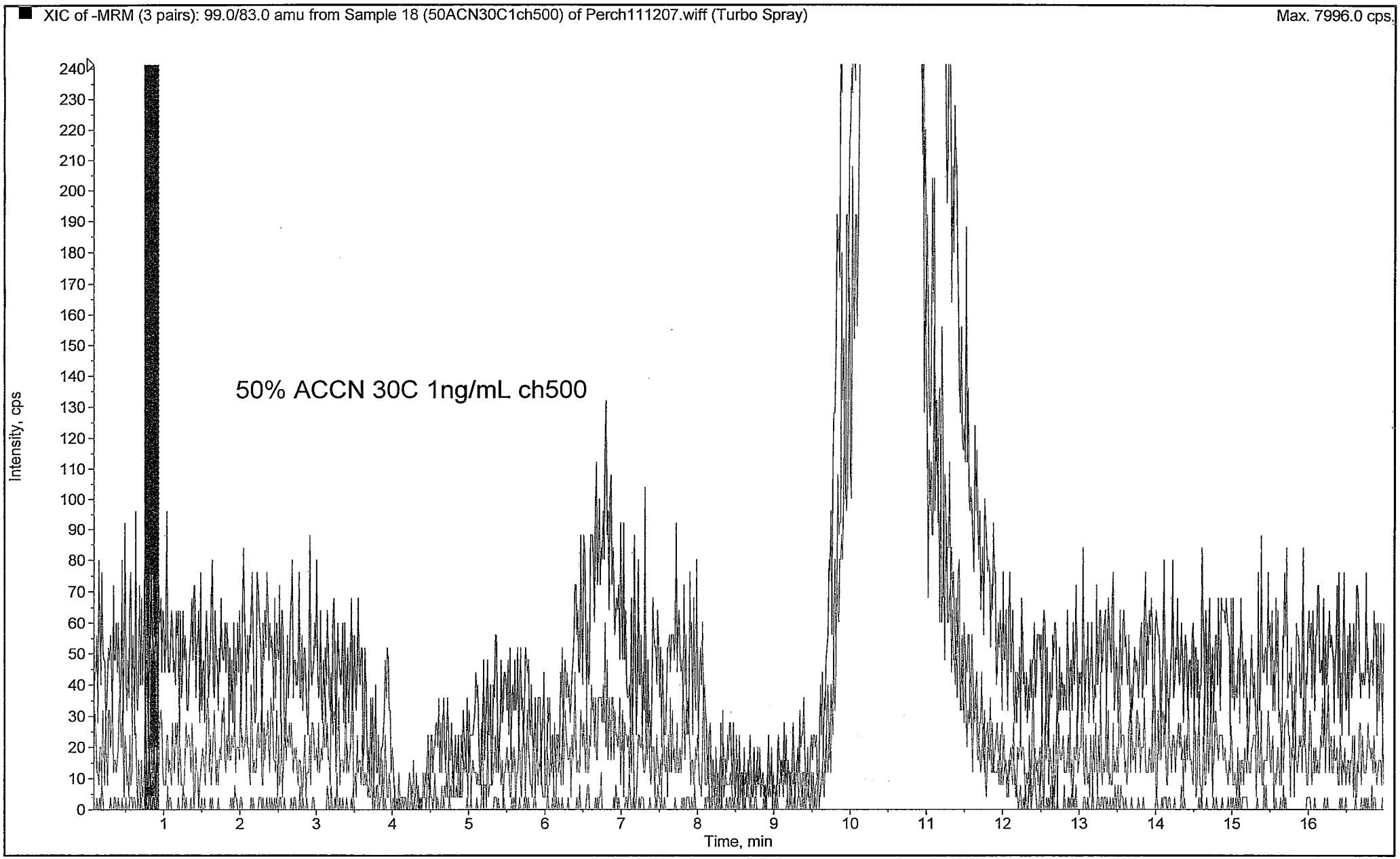

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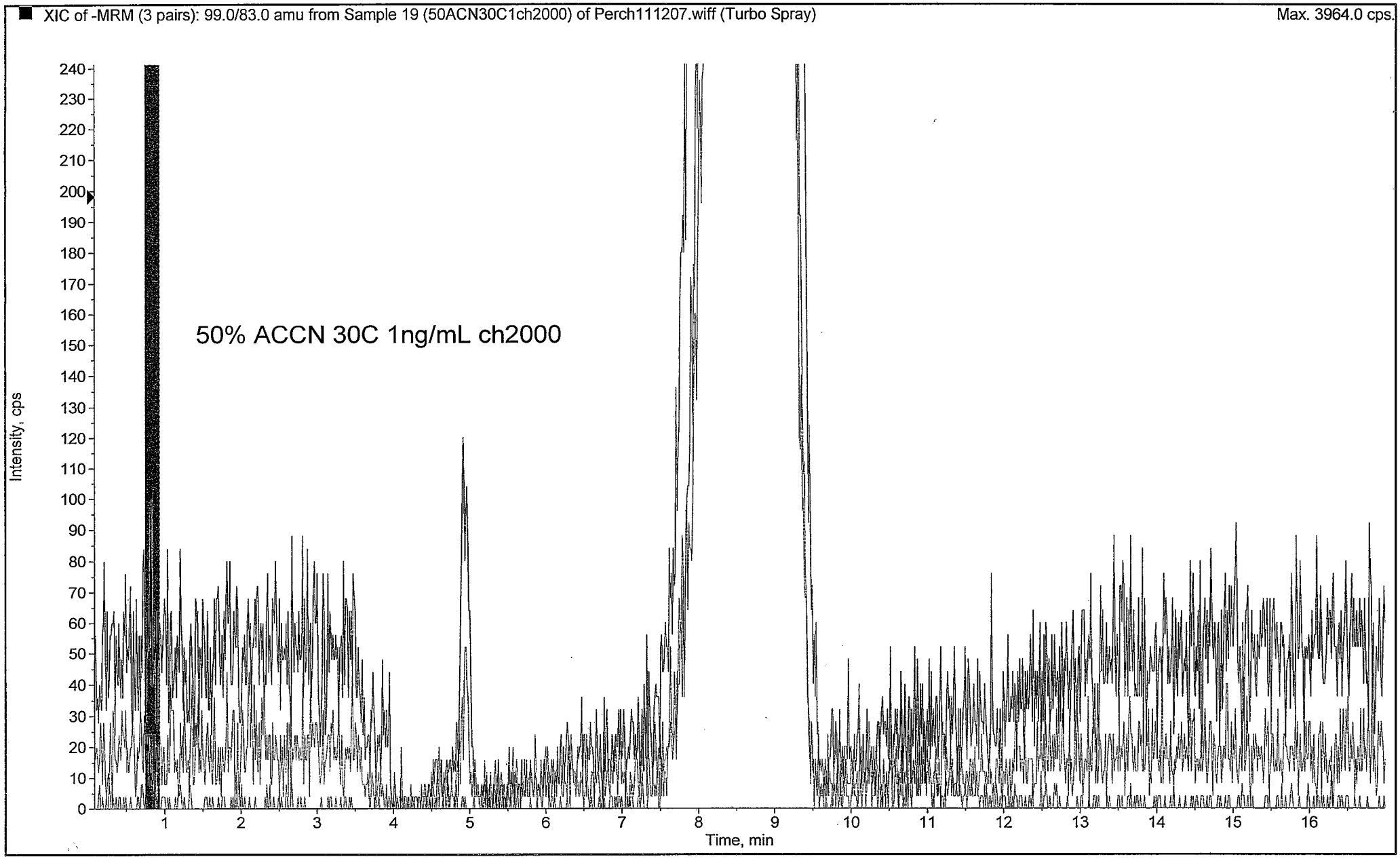
Printing Date: Tuesday, November 13, 2007



Appendix VI

Timeline of Development Activity

Perchlorate Development/API 3200 Timeline 1/11/07

Linda S. Jefferies

10/9/07	Preliminary tuning for Perchlorate with G. Lucus
10/16/07	Tuning Orientation for S. Ignelzi Included DP, EP, CEP, CXP and Source tuning: Temp.. Horizontal and Vertical Probe position,Gas1 and 2, Curtain Gas, and Cad Gas.
10/22/07	Preparation and Tuning of Perchlorate IS: ran perchlorate and IS separately and mixed.
10/23/07	Preliminary Standards ICAL 0.01, 0.05, 0.1, 0.5 and 1ng/mL. Good results, peaks "spikey". Peak shape was later improved via improved acquisition parameters and smoothing.
10/26/07	Tested chromatography while varying ACN concentration in MP. Increasing ACN above 50% did not improve chromatography. Also tested injection volume. Best signal to noise for 75 or 100uL.
10/31/07	New standards for calibration curve. Made intermediate A for 1st and 2nd source and Intermediate B from intermediate A (1st source). Also made challenge controls form 20mg/mL stock from E. Lintner. Stock contained Cl^-, $SO4^-$ and $CO3^-$. Challenge standards containing each 500ppm of each Anion and 100, 0.1 and 0.01ng/mL Perchlorate respectively and Controls containing 0.1 and 0.01ng/mL of Perchlorate. ICAL looks good, but individual peak shape was bad with significant peak splitting. Noticed that after challenge samples were run, peak shapes looked better.
11/1/07	Matrix Matching: MP compounds used with 1.0 STD. Diluted 50:50 with H2O, MP, H2O/ HOAc, and Anion mix. Best result with MP. H2O+ HOAc gave split peak.
11/2/07	Made ACN/HOAc solution (0.1%HOAc) diluted 1:1 with 1 and 0.01 standard. Injected 100, 50, 25 w and w/out IS. Peak shapes good for all 1.0ng/mL stds. IS addition made no difference. 0.01 std not visible.

Concluded that diluting with ACN/HOAc solution gave better peak shape, but dilution of sample with ACN/HOAc should be kept minimal.

11/5/07 Re-optimized source tuning parameters.
Method = PerchMRM110507

CUR	10	new	40
CAD	5	new	10
IS volt	-4500	new	-4000
Temp.	575	new	575
Gas 1	35	new	35
Gas 2	40	new	40

Made new dilutions with 0.5 Std. and added various amounts of ACN/HOAc solution. All injections looked poor. Cleaned front end and tubing connections injections looked better. The 250ul ACN/HOAc +500uL standard was comparable to 500ul ACN/HOAc+500uL STD. Peak shape poor for the samples with 0 or 50uL of ACN/HOAc added.

11/6/07 Determined the optimal amt of ACN/HOAc to add to a given sample volume. All had 500uL of 1ng/mL Std. 500, 250, 100 and 0uL of ACN/HOAc were added respectively. Result, 100ul of ACN/HOAc looks great, 250 and 500 look good, and 0uL of ACN/HOAc looks misshapen. Looked at same concentrations for a 0.01 Std then injected 100, 50, 25 and 10uL.
Also MP made at 0.05% HOAc. Result lower HOAc in MP did not yield better results. It did shift the RT 2 min later. Concluded that original MP is the better choice.

11/7/07 Made mobile phase with the 80% H2O, 20%ACN and 0.1%HOAc and anion challenge samples at 0, 500 and 2000ppm of Anion with a 0.05, 1.0, and a 100ng/mL [Perchlorate]. Results: 20% ACN gave advantage of minimal RT shift with increasing [anion], increased peak splitting also accompanied higher [anion] making it unsuitable for a rugged assay. The 50% ACN MP was more susceptible to RT shift with inc. [anion], but showed less or no peak splitting or ghosting.

11/9/07 Sample with 1ng/mL Perchlorate and either 0, 500 or 2000ppm challenge anions were run in MP with 50%ACN at 20, 30, 40 and 50°C. The 30°C provided the best overall performance including reasonable run time and less peak splitting and ghosting. Although 20°C looked good, the RT for non challenged samples was at 16 minutes which would increase our overall run time by 3 or 4 minutes. Forty and 50°C gave far more ghosting and peak splitting and moved the RT for the 2000ppm anion sample into our intended divert time.

11/12/07 Samples were run using Reserpine MP which is 40% ACN, 30% H2O with 2mm Ammonium Acetate with 0.1% HOAc. The peak shape looked great, but the RT was around 5 min, so our separation from anions would be lost.

11/14/07 New standards were set-up in AS vials including challenge with 1 and 100ng/mL perchlorate and 500 and 2000ppm anion. Samples were tested with the divert acquisition method PerchMRM110507Divert.dam. All challenge samples eluted after the divert. With increasing [anion], RT decreases, and peak shape becomes more asymmetrical and spikey but with valid peak, IS and isotope ratios.

11/15/07 Analyzed samples for Wet Chem. in order to evaluate unusual retention times. 0711111-3, -7, -19, -39, -31, 071112-4, and 0710285-1. Data returned to Wet Chem. Helped confirm their suspicions regarding false positives.

11/20/07 Re-optimized front end after speaking w/Chris Borton.
New settings: Gas 1 55
Gas 2 75
CAD 12
Curtain Gas 25
Temp 575
Also increased scan time in order to smooth chromatograms by acquiring fewer scans/peak. Increased form 250 to500 msec. (later increased to 750msecs.) This gives around 20 scans/peak as per Chris Borton.

11/27-28/07 New standards were made from Source 1 and 2 these were subsequently entered into the standards database (ST071203-x). ICV and CCV controls were also made. In addition, solutions were made for the ITS interference threshold study which included 1.0ng/mL standards at: 500, 2000, 4000 and 6000 ppm. All ITS standards had good recoveries however, the IS for the6000ppm sample showed ion suppression therefore new standards (4500, 5000 and 5500ppm) were made in order to pinpoint the threshold. These standards showed that ionization suppression level (the level where IS area is less than 50% of normal) is at 5000ppm. Since we are planning to use the divert in order to protect our instrument, we will set the MCT (Matrix Conductivity Threshold) at 2000ppm. Again, quantitation was accurate even for the 6000ppm standard which showed great suppression. Conductivity measurements on these standards was later conducted by SI.

11/29/07 Analyzed preliminary MDL standards at 0.002ng/mL. SI made soil trials at 0.002, 0.005 and 0.01ng/mL (final in 10ml H2O). The water trials at 0.002 were below the Y intercept so they were too low to use. Note: I tried a shooter made form MP stored in a glass vial (black screw cap). The resultant peak looked as if it were contaminated with anion mix. The MP was discarded, the old shooter was ok so this was not a chromatography effect ie: dirty column, etc.

11/30/07 Tried MDL trials at 0.05ng/mL. Difficult to quantitate. SI made Soil MDL @ 0.02ng/mL (actually determined to be 0.01ng/mL) in final volume these were not used. Soil MDL's @ 0.02ng/mL were remade by SI on 12/03/07.

12/03/07 Made new ICV and CCV to compare to old.

12/04/07 Made new MB, 2MDL trials at 0.01ng/mL and a new Std at .005ng/mL. Set up and ran SI soil MDLs at 0.02ng/mL in water extract and new water MDLS at 0.01ng/mL.

12/05/07 Made a new confirmatory MDL at 0.01ng/mL (not used). The 0.005ng/mL standard was not used as a Std and so was later used as a confirmatory MDL.

12/10/07 Determined that the difficulty with low level quantitation might be due to the fact that we had stopped running the 0.5 and the 5ng/mL standard. A 0.5ng/mL standard was made along with new MP.
NOTE: I noticed that MP that had been stored in a small glass Kimble Opticlear vial for diluting the shooter again appeared contaminated. Since the MP had been stored in this vial for several days, I concluded that a substance on or in the glass must be contaminating the MP stored in these vials causing a distorted peak shape.
ICAL vials were prepared along with a CCB and Millipore water form Metals. The CCB and the Millipore water did not have IS added (at that time the CCB was prepared as a method blank). In reviewing the ICAL, the 50 and 100ng/mL standards looked off.

12/11/07 New ICAL standards were made @ 50ng/mL and 100ng/mL . The PE sample 0711245-1 was diluted as per instructions from client. The following controls: LODV @ 0.02ng/mL, ICS @ 0.05ng/mL with 2000ppm each anion, LCS (for water only) 0.05ng/mL (re-made on 12/12/07) MS 2mL sample 0711043 spiked at 0.05ng/mL, and MB. Result Sample @9.56ug/L acceptable.

12/18/07 Looked at each isotope and IS in order to provide tuning print-outs for data packages.

12/19/07 Performed LCS experiment to prove competency for soil sample preparation.

12/20/07 Perchlorate analysis for LANL (WO# 0711245)

Appendix VII

Summary of Testing Requirements

Summary of Testing Requirements

Client Requirements Used to Create Program Specifications and Nicknames

Perchlorate Criteria – Data Acquisition and Review Criteria

Parameter	**EPA 6850**[1] **(Paragon Std.)**	**DOD Handbook Rev. 1 Change 1**[2] **(DOD Program Spec. and Nickname)**	***Winkler, Willey, Minteer***[3] **(LANL ClO4 Program Spec. and Nickname)**
Reagent Blank	N/A System "interference free doc. By MB (9.3)"	Prior to calibration, after over-range samples; < ½ RL	ICB, CCB and MB are specified (see below)
ICAL	r= > 0.995 min. 6x plus blank (see MB)	r= > 0.995; ≥ 5 std.s RSD < 20% ea. Linear Fit-No Force Conc. = [Y-inter.] ≤ LOD	"blank and 5 points" r= > 0.995 Is 24hr. freq. required?-asked... Conc. = [Y-inter.] < 50% DL (DL=SN m/z85 > 25:1)
ICV (alt. Source)	± 15% (conc. within cal. range)	± 15% (mid-range conc.)	± 15% (mid-range conc.)
Matrix Challenge **(Initial Study)**	Initial study, Determine MCT CL – screen, dilute	1x/20 samp. Batch same pretreat as samp.s Conc. at RL Result ± 30%	Do cleanups, necessary for precision of ion ratio, control chart ion ratio.
ICB **(post ICV, with IS)**	N/A	N/A	Post ICV < DL
CCB **(post CCV, with IS)**	N/A	N/A	Post CCV < DL
Field Samples	Prep. Batch of 20	Prep. Batch of 20	Prep. Batch of 20
ICS (ICSL)	N/A	1x/20 samp. Batch same pretreat as samp.s Conc. @ RL Result ± 30%	500ppm (4 salts) @ PQL; about 4x DL ± 20%
CCV	After 10 field samples and end of seq. Alt. LL and mid conc.; 15% ML; ±50% LL (LL = LODV[3] level)	Mid-level after 10 samples; ± 15% (field samp.s or injections-ask Wiley)	± 15%
MB	@ reagent changes and with each batch; < LLOQ	One per batch, < ½ RL	One per 20 sample preparation batch, < DL; Report results >10x blank-only. RE if interference
LCS	80-120%	1x/20 @RL 80-120%	80-120% Re-analyze associated samples and LCS if out

LLOQ[1] – LODV[2] – DLV[3]	Low point in curve or RL standard, ± 50%, may use low range CCV (at end)	LODV: Bracket Batch Spk. @ 2x LOD Rec. ± 30% LODV Failure: at > LOD < RL, RE samp.s in range; OK samp.s above, RE fails=Q flag	DLV-bracket anal. run RL S/N m/z 85 ≥ 25:1 DL std. ± 30%
MS	≥1per prep. batch 80-120%H2O 70-130% Soil	≥1per prep. batch @RL (impractical?/SI) 80-120%	75-125% N/A if sample 4x spike Analytical spike req.
Field Sample Dup. or MSD	*RPD < 15%*	≥1per prep. batch RPD < 15%	1x per batch (≤20 field samples) RPD < 20% < PQL and > DL then ± DL <DL then NA for RPD crit.
RT Window Study	Not necessary, IS identifies; 0.2min max. RT error vs. IS	Not Defined Assumed = 6850	Not Defined Assumed = 6850
Isotope Area Ratio (m/z 83/85)	Isotope Ratios: m/z 83 area/85 area = 2.3-3.8 (vs. theoretical) or ± 30% vs. mid-range standard or CCV if other day	Every sample, QC samp. and standard Theor. 3.06; must fall 2.3-3.8 (25% window)	Statistically derive as additional acceptance criteria for ClO_4 ID. Mean ratio pop. ± 10% vs. 3.08 theor. or standards (tele) s < 0.2 guideline Same window around system bias ratio OK per Minteer –see telephone record/SI
IS Recovery (IS = IRCS[1])	± 50% rec.	± 50% IS rec. RRT 83/89 = 1.0 ± 0.02	Recovery ± 50% If out, dilute until met (cleanups expected)
Cleanup **(as needed)**	Optional C18 Matrix Div. Opt. "Can be used"		2x Ba2+, 1x H Cartridge

Acquisition Sequence (EPA 6850)

- ICAL (6 or more points, i.e. 0.01-100 µg/L); we use 9.
- ICV Alternate source standard, acceptance ± 15% [1,2,3]
- Sample Batch QC: MB, LCS, MS, MSD or Sample Duplicate (Dup)
- Field Samples: ≤ 10 field samples
- CCV After ever ten samples[2], mid-level, ± 15%
- Sample Batch QC: MB, LCS, MS, MSD or Sample Duplicate (Dup)

- Field Samples: ≤ 10 field samples
- CCV Brackets samples (LL CCV, rotate vs. mid-level)
- Sample Batch QC: MB, LCS, MS, MSD or Sample Duplicate (Dup)
- Field Samples: ≤ 10 field samples
- CCV Brackets samples. Final CCV is low level (LL); (LODV = CCV_{LL}) or may follow CCV with LODV
- **NOTE:** The LODV meets the 6850 criteria for a LL CCV (LODV is within ICAL range), therefore, the LODV may be used as the CCV.
- LODV ± 50% [1] We run 2x MDL (as determined per [3]) level. Satisfies 6850[1] requirement also.

Acquisition Sequence (EPA DOD Perchlorate Handbook Rev. 1 Change 1)

- Reagent Blank Prior to calibration, after overrange samples and after each batch, <1/2 RL
- ICAL (6 or more points, i.e. 0.01-100 µg/L); satisfies [1,2,3]
- ICV Alternate source standard, acceptance ± 15% [1,2,3]
- LODV ± 30% [2,3] Approximately 2x MDL level.
- ICS ≥ 500ppm (4 salts), @ PQL, 4x DL, ± 20%[3] (prepare one per 20 sample prep. batch; same prep. and cleanups as other samples in batch), verifies method performance at the MCT.
- Sample Batch: MB, LCS, MS, MSD or Sample Duplicate (Dup), 10 samples
- CCV After every ten samples[2], mid-level, ± 15%
- LODV ± 30% [2,3] Approximately 2x MDL level, brackets each batch of ≤20 field samples
- 10 samples (injections), continue sample groups (plus QC) until close of run.
- CCV Brackets samples
- LODV ± 30% [2,3] Approximately 2x MDL level, brackets each batch of ≤20 field samples
- Reagent Blank Prior to calibration, after overrange samples and after each batch, <1/2 RL

Acquisition Sequence (Winkler, Wiley, Minteer-LANL)

- ICAL (6 or more points, i.e. 0.01-100 µg/L); satisfies [1,2,3], daily!??!
- ICV Alternate source standard, acceptance ± 15% [1,2,3]
- ICB Blank with IS; <DL (DL = adjusted MDL)
- LODV ± 30% [2,3] We run 2x MDL level. Should meet 6850[1] also
  Note: EPA 6850 requires LLOQ, bottom of curve ± 50% so, this LODV check is more rigorous and more frequent.
- ICS ≥ 500ppm (4 salts), @ PQL, 4x DL, ± 20%[3] (one per 20 sample batch)
- 10 Analytical Samples (injections) MB, LCS, MS, MSD or Sample Duplicate (Dup)
- CCV After ever ten analytical samples (injections)[2], mid-level, ± 15%
- CCB Blank with IS (not filtered, otherwise it's an MB)
- LODV as above, brackets each batch

- 10 analytical samples, continue sample groups (plus QC) until close of run.
- CCV Brackets samples
- CCB Follows CCV (not required in 6850 or DOD Handbook)
- LODV Bracket batch[3]
- ICS 1 per batch (see above) of 20 ± 15%[2], 500ppm (or higher) @ RL ± 15%[3]
- CCB Free of significant interference

NOTE: DL is an "adjusted MDL (not per 40CFR Part 136 Appendix B); DL = minor ion S/N ≥ 25:1. We use 0.01 µg/L, RL 0.05µg/L

References

[1]EPA Method 6850; "Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MA/MS)"
Rev. 0, January 2000

[2]DOD Perchlorate Handbook Rev. 1 Change 1

[3]"Proposed Calibration and Quality Control Criteria for Perchlorate Analysis by LC/MS and LC/MS/MS" by Dr. Paul Winkler, Janice Willey and Mark Minteer (provided by Mark Minteer, not published)

Appendix VIII

Performance Testing Result

PERFORMANCE EVALUATION

First Choice for Quality | RTC

Off-study
WP07-4-126

2-Nov-2007 through 13-Dec-2007

RT2541
RTC Labcode

CO00078
US EPA Labcode

Paragon Analytics
Deb Scheib
225 Commerce Drive
Fort Collins CO 80524

Thank you for participating in study WP07-4-126. Additional information about this study may be found online at www.rt-corp.cor
If you have any questions or comments about this study please contact me.

Sincerely,

Christopher Rucinski
Quality Director

2931 Soldier Springs Road
Laramie, WY 82070
(307) 742-5452
www.rt-corp.com

ACCREDITED
Certificate # 2122.01

Dataset

Method One

Accreditors

Evaluations of this dataset will be sent to the accreditor(s) listed below using your laboratory's labcode listed above each accrediting agency. If any of the information listed below is incorrect, please contact RTC immediately.

Accrediting Labcode CO00078
California Dept. of Health Services
Environmental Lab Accred. Program Branch

104 Fred Choske
850 Marina Bay Parkway
Bldg. P, 1st Floor, MS 7103
Richmond CA 94804
UNITED STATES

Accrediting Labcode CO00078
Connecticut Dept. of Public Health

350 Jeffrey Curran
410 Capital Ave.
P.O. Box 340308
MS#51 - Lab
Hartford CT 06134
UNITED STATES

Accrediting Labcode CO00078
DoD EDQW
Laboratory Quality & Accreditation Office

949 Bill Ingersoll
NAVSEA Programs Field Office
1661 Redbank Rd
Bldg 302, Rm 209
Goose Creek SC 29445
UNITED STATES

Accrediting Labcode CO00078
Florida DEP Environmental

359 Silkie Labie
2600 Blair Stone Road
Tallahassee FL 32499
UNITED STATES

Accrediting Labcode CO00078
Louisiana Dept. of Environmental Protection
OMF - Lab Accreditation

112 Karen Varnado
PO Box 4314
Baton Rouge LA 70821
UNITED STATES

Accrediting Labcode CO00078
Nevada Division of Env. Protection

366 Sara Rairick
901 S Stewart Street
Ste 4001
Carson City NV 89701-5249
UNITED STATES

Accrediting Labcode CO00078
New Jersey DEP

353 Rachel Werner
9 Ewing Street
PO Box 424
Trenton NJ 08625-0424
UNITED STATES

Accrediting Labcode CO00078
North Dakota Department of Health

119 Errol Erickson
Chemistry Division
2635 E. Main Ave.
PO Box 937
Bismarck ND 58502-0937
UNITED STATES

Accrediting Labcode CO00078
Oklahoma Dept. of Environmental Quality
Laboratory Certification
121 David Caldwell
P.O. Box 1677
Oklahoma City OK 73101
UNITED STATES

Accrediting Labcode CO00078
Texas CEQ

384 Frank Jamison
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Austin TX 78711-3087
UNITED STATES

Accrediting Labcode CO00078
Utah Bureau of Laboratory Improvement

215 Lab Accred.
46 N. Medical Drive
Salt Lake City UT 84113-1105
UNITED STATES

Accrediting Labcode CO00078
Washington State Dept. of Ecology
QA Section
129 Connie Schreiber
Certification
PO Box 488
Manchester WA 98353
UNITED STATES

Miscellaneous Analytes

Analysis
EPA 6850
HPLC-MS-MS

Method Number 10304606
Technology Code HPLC-MS-MS

	Result Units	Accept / Warn	Z	Evaluation

Miscellaneous Analytes (continued)

(continued)

Analysis
EPA 6850
HPLC-MS-MS

Method Number 10304606
Technology Code HPLC-MS-MS

	Result Units	Accept / Warn	Z	Evaluation
Perchlorate [4] 1895 / I-266 - Lot 011165	9.56 µg/L	6.82 to 10.2 7.38 to 9.66	1.83	**Acceptable**

End of Method One

Dataset

Method Two

Accreditors

Evaluations of this dataset will be sent to the accreditor(s) listed below using your laboratory's labcode listed above each accrediting agency. If any of the information listed below is incorrect, please contact RTC immediately.

Accrediting Labcode CO00078
California Dept. of Health Services
Environmental Lab Accred. Program Branch

104 Fred Choske
850 Marina Bay Parkway
Bldg. P, 1st Floor, MS 7103
Richmond CA 94804
UNITED STATES

Accrediting Labcode CO00078
Connecticut Dept. of Public Health

350 Jeffrey Curran
410 Capital Ave.
P.O. Box 340308
MS#51 - Lab
Hartford CT 06134
UNITED STATES

Accrediting Labcode CO00078
DoD EDQW
Laboratory Quality & Accreditation Office

949 Bill Ingersoll
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Goose Creek SC 29445
UNITED STATES

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Florida DEP Environmental

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UNITED STATES

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OMF - Lab Accreditation

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Accrediting Labcode CO00078
Nevada Division of Env. Protection

366 Sara Rairick
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Ste 4001
Carson City NV 89701-5249
UNITED STATES

Accrediting Labcode CO00078
New Jersey DEP

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UNITED STATES

Accrediting Labcode CO00078
North Dakota Department of Health

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UNITED STATES

Accrediting Labcode CO00078
Oklahoma Dept. of Environmental Quality
Laboratory Certification
121 David Caldwell
P.O. Box 1677
Oklahoma City OK 73101
UNITED STATES

Accrediting Labcode CO00078
Texas CEQ

384 Frank Jamison
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UNITED STATES

Accrediting Labcode CO00078
Utah Bureau of Laboratory Improvement

215 Lab Accred.
46 N. Medical Drive
Salt Lake City UT 84113-1105
UNITED STATES

Accrediting Labcode CO00078
Washington State Dept. of Ecology
QA Section
129 Connie Schreiber
Certification
PO Box 488
Manchester WA 98353
UNITED STATES

Miscellaneous Analytes

Analysis
EPA 331.0
HPLC-MS-MS

Method Number 9995454
Technology Code HPLC-MS-MS

	Result Units	Accept / Warn	Z	Evaluation

Miscellaneous Analytes (continued)

(continued)

Analysis
EPA 331.0
HPLC-MS-MS

Method Number 9995454
Technology Code HPLC-MS-MS

	Result Units	Accept / Warn	Z	Evaluation
Perchlorate [4] 1895 / I-266 - Lot 011165	9.56 µg/L	6.82 to 10.2 7.38 to 9.66	1.83	**Acceptable**

End of Method Two

Sample Information

Perchlorate

PEI-266

Study Lot 011165
Mfg Lot 011165

	Units	Proficiency Value	Proficiency Std. Dev.	Mean	Standard Deviation	Robust Mean	Robust Std. Dev.	Gravimetric
Perchlorate 1895 Miscellaneous Analytes	µg/L	8.52	0.57					8.52 ± 0.082

Program analyte accrediting footnotes

[1] NELAC
[2] EPA
[3] Other
[4] A2LA
[5] NELAC Experimental

STANDARD OPERATING PROCEDURE 448 REVISION 1

TITLE: **DETERMINATION OF PERCHLORATE IN LIQUIDS AND SOLIDS USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY, ELECTROSPRAY IONIZATION TANDEM MASS SPECTROMETRY (LC/MS/MS)**

FORMS: **415** **(use current iteration)**

APPROVED BY:

TECHNICAL MANAGER _____ DATE 1-5-09

QUALITY ASSURANCE MANAGER _____ DATE 1/5/09

LABORATORY MANAGER _____ DATE 1/5/09

HISTORY: NEW Rev0, 3/20/08; Rev1, 12/31/08.

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references – EPA SW6850 (multi-matrix), DoD Perchlorate Handbook, and Method 331.0 (drinking water matrix), are used to determine the concentration of perchlorate in liquid and solid samples.

Analyte	CAS #
Perchlorate ClO_4^-	14797-73-0

2. SUMMARY

Water samples are filtered prior to analysis, soil samples are extracted with reagent water then filtered prior to analysis. An internal standard (IS) is added to the sample/extract, which is then well-mixed, before being filtered and analyzed. Samples/extracts may be subjected to a C_{18} or ion exchange cleanup prior to HPLC separation with subsequent multi-reaction monitoring (MRM) analysis by LC/MS/MS, with electrospray ionization and isotope dilution quantitation. Data is captured via an integrator, and instrument operation and data processing are assisted by a personal computer (PC).

NOTE: Cleanup removes high hydrocarbon level interferences and discoloration. The ion chromatography versions of this method use multiple cleanup procedures via filter technology, such as Hydronium (–H) for carbonate, silver (-Ag) for chloride, and barium (Ba^{2+}) for sulfate. Method validation showed that such cleanup is usually unnecessary when using the K' column and solvent diversion valve. Two barium cartridges with one hydronium cartridge in parallel or a combined bed cartridge may be used to remedy extreme matrix challenges.

A 100uL sample (including added acetonitrile and acetic acid) is introduced into the

HPLC. Perchlorate (ClO_4^-) is separated chromatographically from the sample/extract matrix. Early eluting interferences may be diverted to waste prior to introducing the HPLC stream to the ion source. The HPLC is connected to the ion source of the MS/MS, where electrospray ionization is performed. Perchlorate ions m/z 99 ($^{35}ClO_4$), 101($^{37}ClO_4$) and 107 ($^{35}Cl^{18}O_4$) are selectively filtered to the collision cell by the first quadrupole mass filter, where perchlorate is partially fragmented via collisionally induced dissociation. Multiple reaction monitoring (MRM) is performed and fragment ions m/z 83 ($^{35}Cl0_3^-$), 85 ($^{37}ClO_3^-$) and 89 ($^{35}Cl^{18}O_3^-$) are selectively detected. Detector responses are processed by an electronic integrator and the result is determined using an internal standard quantitation calculation. Since the internal standard used is an isotopically labeled version of the analyte and is added at the start of the sample preparation process, this is an isotope dilution method with recovery correction included.

This technique is highly sensitive and selective. Perchlorate presence, confirmed by MS/MS MRM products, has a high level of certainty.

Precursor Ion (m/z)	**Fragment Lost (m/z)**	**Product Ion (m/z)**
$^{35}ClO_4$ (99) quant. ion	^{16}O (16)	$^{35}Cl0_3^-$ (83)
$^{37}ClO_4$ (101) conf. ion	^{16}O (16)	$^{35}Cl0_3^-$ (85)
$^{35}Cl^{18}O_4$ (107) IS ion	^{18}O (18)	$^{35}Cl^{18}O_3^-$ (89)

Please note that as stated in Section 10, the requirements prescribed in this SOP meet or exceed the requirements set forth in EPA SW6850. A summary of QC parameters and associated corrective actions is provided as a Table at the end of the SOP. Note also (Section 3.2) that these requirements may be superceded by the requirements prescribed via a specific client program specification. Consult the individual program specification for client-specific criteria and corrective actions.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review. Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.2 Paragon's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede Paragon's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for

client-specific requirements prior to initiating handling of samples or data.

3.3 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the benchsheet indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work, and documentation of measures taken to remediate the data.

3.4 It is the responsibility of all personnel who work with samples involving this procedure to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented as prescribed in SOP 928.

4. INTERFERENCES

4.1 Sulfate has essentially the same parent mass as perchlorate. However, sulfate cannot form the collision products that are monitored from perchlorate, and sulfate is well separated from perchlorate by the K' RP column.

4.2 Carbonate, chloride and other anions and eluting ions may also interfere if present at sufficiently high concentrations.

4.3 Co-eluting entities can cause ion suppression. Absence of ion suppression as a problem below 4500mg/mL challenge (31,200µS/cm) was demonstrated during validation of EPA Method 6850 and is documented as shown in Table 3 below:

Table 3
Matrix Challenge Study Results

Conc. Suppressors (mg/mL)	Conductivity (µS/cm)	Suppression Observed	RT Drift
500	5000	No	Within Divert
2000	16620	No	Within Divert
4000	28500	No	Exceeds Divert
4500	31200	No	Exceeds Divert
5000	34000	IS < 50%	Exceeds Divert
5500	36700	IS < 50%	Exceeds Divert

NOTE: $^{35}Cl0_3^-$ (m/z83)/ $^{35}Cl0_3^-$(m/z85) ratio acceptable throughout study range.

A study to determine the maximum tolerable concentration of known ion suppressors bicarbonate, carbonate, chloride and sulfate was performed. This study included no divert event. Diversion of the HPLC effluent prior to elution of perchlorate may be used to protect the instrument from contamination. However,

as sample conductivity increases, perchlorate retention decreases, and perchlorate will go to divert at conductivity beyond about 17000 µS/cm. For this reason, 16620uS/cm (2000 mg/mL) is the level selected for the matrix conductivity threshold (MCT).

In light of retention time drift, samples with sufficiently high conductivity to cause perchlorate to be diverted from the mass spectrometer must be diluted for analysis (assuming required reporting limit can be met), subjected to cleanup, or analyzed without divert. Dilution may be performed routinely, provided that the required sensitivity is achieved.

An Interference Check Sample (ICSL), may be analyzed to verify method performance for perchlorate, at the RL, in the MCT solution. Refer to QC Table at the end of this SOP or information given in the specific client program specification for ICSL acceptance criteria.

4.4 Additional cleanup procedures may be used. SPE C_{18} (for organic contamination) and/or cartridge cleanups such as Ba^{2+} (for sulfate), -H (for carbonate), and Ag (for chloride), may be used if removal of these interferences is appropriate. Two barium and one hydronium cartridge in series have been shown to be effective in removal of high sulfate and carbonate interferences. If cleanup columns are used, associated QC samples must be prepared in the same manner as the batch sample(s).

4.5 Interferences are also minimized by the use of high purity reagents.

5. APPARATUS AND MATERIALS

5.1 HPLC, AUTOSAMPLER, DETECTORS

Shimadzu Prominence (or equivalent) binary HPLC equipped with:

- gradient pumping system
- autosampler
- system controller – interface to MS data system
- column oven

Tandem mass spectrometer

- Applied Biosystems API 3200 MS/MS or equivalent

5.2 DATA ACQUISITION

Any data acquisition system capable of acquiring, storing and processing HPLC/ mass spectrometry data (e.g., Analyst™ or equivalent) to support qualitative and quantitative requirements may be used.

5.3 COLUMNS - Equivalent columns may also be used

Guard Column: Optional; guard must not undermine system performance
Analytical Column: K' Technologies, Inc. RP HPLC 4x250mm KPRPP-X250

5.4 MEASURING DEVICES

- Pipettes – air displacement/conical tip, or equivalent, various µL ranges; operated as prescribed in SOP 321
- Volumetric Flasks (Class A) – various sizes
- Graduated cylinders – 5 to 1000mL glass or polymer (translucent plastic, PMP), or equivalent
- Analytical Balance (for preparation of standards and matrix challenge solution); operated as prescribed in SOP 305
- Top-Loader Balance: to 0.01g (for sample aliquot prior to extraction extraction) ; operated as prescribed in SOP 305
- VWR Conductivity Meter Model 23226-523 or equivalent

5.5 CONSUMABLES

- Filter for Mobile Phase: Filter the mobile phase using vacuum filtration with a 0.45µm nylon membrane filter; 47mm (Whatman 7404-004) or equivalent
- Disposable 50mL centrifuge tubes, Falcon #35720 or equivalent
- Disposable transfer pipettes, SAMCO #202 or equivalent
- Syringe Filter, Restek 25mm, 0.22µm PTFE #26146, 0.45µm or equivalent
- Autosampler vials and caps
- C_{18} Solid Phase Extraction (SPE) Cartridges (optional cleanup)
- Ion exchange cartridges: Ba^{2+}, –H and/or Ag (optional cleanup)

NOTE: **High Density Polyethylene** (HDPE) instead of glass is acceptable for perchlorate per EPA 331.0.

6. REAGENTS - All reagents must be HPLC grade or better

6.1 Mobile Phase: 50:50 Acetonitrile (ACN): HPLC H_2O, with 0.1% Acetic Acid (HOAc) – Make by mixing 500mL HPLC reagent water and 500mL ACN, add 1.0mL of acetic acid. Filter as per Section 5.5.

6.2 Internal Standard (IS) Stock Solution
Isotopically labeled perchlorate ($^{35}Cl^{18}O_4$) as neat salt or in HPLC water (concentration as received from manufacturer)

6.3 Working Internal Standard Solution (WIS)
Typically the concentration of the WIS is 50µg/L of isotopically labeled perchlorate ($^{35}Cl^{18}O_4$) in HPLC water (final IS concentration in samples is

0.5µg/L). The absolute concentration is not critical as long as all samples and standards are spiked equivalently.

6.4 Acetonitrile/Acetic Acid Solution: ACN + 0.1% HOAc.

6.5 Solid phase column activation (optional cleanup)
Methanol, HPLC grade

6.6 Ottawa sand

6.7 STANDARDS
All standards are maintained per SOP 300. In the event of a conflict, the specific guidance in this SOP shall supercede that of SOP 300. All stock and intermediate standards are documented in the laboratory's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Certificates of Analysis from vendor materials are maintained by the applicable laboratory Department.

6.7.1 At minimum, two independent sources of target analyte are required. First source materials are used to create calibration, continuing calibration verification (CCV) and QC sample spike standards. Second source materials are used to create the initial calibration verification (ICV) solution (used to independently verify the accuracy of the initial calibration, ICAL).

6.7.2 An appropriate volume of commercial stock standard is diluted to a specific volume to create both Intermediate and Working Standards; these dilutions are shown below. Perform all dilutions using calibrated pipettes (SOP 321). Do not use standards beyond their expiration date. EPA 6850 specifies a one-year expiration period for intermediate perchlorate standards. This applies to labeled internal standard as well as native perchlorate standards.

Intermediate standards are prepared from 1000µg/mL perchlorate stock (first source for calibration standards, second source for ICV) as follows:

Intermediate Perchlorate Solution A:
500µg/L (50µL of 1000µg/mL perchlorate to 100mL HPLC water)

Intermediate Perchlorate Solution B:
5µg/L (100µL of A to 10mL HPLC water)

TABLE 4
CALIBRATION STANDARDS

Level	Analyte Conc. (µg/L)	Standard Diluted (Intermediate Solutions Above)	Standard Added (µL)	Final Volume (mL)
ICAL 9	100	A: 500 µg/L Stock	2000	10.0
ICAL 8	50	A: 500 µg/L Stock	1000	10.0
ICAL 7	10	A: 500 µg/L Stock	200	10.0
ICAL 6	5.0	A: 500 µg/L Stock	100	10.0
ICAL 5	1.0	B: 5.0 µg/L	2000	10.0
ICAL 4	0.5	B: 5.0 µg/L	1000	10.0
ICAL 3	0.1	B: 5.0 µg/L	200	10.0
ICAL 2	0.05	B: 5.0 µg/L	100	10.0
ICAL 1	0.01	B: 5.0 µg/L	20	10.0
ICV	1.0	B: 5.0 µg/L (2nd source)	2000	10.0

NOTE: Alternate equivalent dilution schemes may be used as appropriate.

NOTE: The ICV is to be utilized at the midpoint of the calibration curve. Based on a logarithmic (i.e., order-of-magnitude) view of the calibration, the ICV has currently been established as 1.0ug/L, which is two orders of magnitude above the low standard and two orders of magnitude below the high standard. This interpretation provides for the best quantitation of perchlorate, particularly because it retains focus at a concentration level most likely to be of primary interest regarding environmental perchlorate samples.

6.8 Interference Check Solution Stock (ICSS) 20mg/mL of each ion: Cl^-, SO_4-, CO_3^-, and $HCO_3^=$. Make by weighing 3.30g NaCl; 2.96g Na_2SO_4; 3.53g Na_2CO_3; and 2.76g $NaHCO_3$ and bring to 100mL with HPLC water. Mix thoroughly, then filter solution through a 0.45µm filter.

6.9 Interference Check Standard for LC/MS (ICSL). For 2000ppm of each anion and 0.05µg/L perchlorate, add 1mL ICSS and 100µL Intermediate Perchlorate Solution B (5µg/L) and bring to 10mL with HPLC water.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Water samples are collected in 125mL sterile HDPE bottles; solid samples are collected in a 4oz amber glass jar. Headspace (one third of total volume) **should** be present to minimize anaerobic biodegradation.

7.3 Store samples at 4±2°C.

7.4 Liquid samples must be analyzed within 28 days of collection. Extracts must be generated from solid samples, within 28 days of collection; the extracts must then be analyzed within 28 days thereafter. A Q flag is appended if samples are out of holding time when received by the laboratory.

8. PROCEDURES

8.1 TYPICAL SYSTEM OPERATING CONDITIONS

HPLC DEVICE

Flow Rate (mobile phase):	0.5 mL/min
Auto Sampler Temperature	15°C
Injection Volume:	100 µL
Column Temperature:	30°C
Run Time:	16 min. (2 min. beyond peak tail)
Ionization Mode:	Turbo Spray (ESI)
Polarity:	Negative

8.2 LCMS-MS MAINTENANCE

Prior to analyzing samples or establishing calibration curves, the following suggested maintenance may be performed/documented to aid in achieving more consistent results:

- Change the column frits as needed (i.e., if pressure increase is observed).
- Ensure that mobile phase provides expected chromatography for perchlorate.
- Flush the entire analytical system and back-flush the column with 50:50 ACN:H2O after each use with this method.
- Clean the K' column by pumping 90:10 ACN:Water. Store the column in the same solution.

  NOTE: The column should be flushed with cleaning solution every 2 months if storing for longer than 2 months.

- Clean or change inlet or outlet valves as appropriate to maintain acceptable flow and accurate flow rate.
- Clean the curtain plate. **CAUTION - source housing and curtain plate may be extremely hot!** Remove the ion source (not necessary to break vacuum); carefully remove the curtain plate and clean on a dust-free surface. Use a dust-free wipe with HPLC water and methanol or 1:1 HPLC

water:ACN with 0.1% formic acid. Carefully reassemble.

8.3 INSTRUMENT MASS CALIBRATION AND TUNING; ANALYTE OPTIMIZATION

The LCMS-MS must have valid mass calibration and tuning, and analyte optimization prior to the performance of any sample analysis.

8.3.1 Mass Calibration and Tuning

Mass calibration ensures that mass peaks are assigned the correct mass-to-charge values. Tuning is the adjustment of the instrument's resolution off-sets to ensure the best peak response. Mass calibration and tuning are performed per manufacturer's instructions.

- The calibration is updated on an as needed basis (i.e., when QC failures are observed, ion masses show large deviation from known masses, major instrument maintenance is performed, or if the instrument is moved).
- If problem is suspected, check calibration with polypropylene glycol (PPG) infusion per manufacturer's recommendation. Adjust as necessary or appropriate.
- Acceptance Range: 0.7 +/- 0.1 (unit resolution)

8.3.2 Analyte Optimization - Analyte optimization considerations consist of both Compound Dependent Parameters and Source Dependent Parameters.

<u>Compound Dependent Parameters</u>:

Declustering Potential (DP)
Entrance Potential (EP)
Pressure of the collision gas during MS/MS scan (CAD gas)
Collision Energy (CE)
Collision Cell Entrance Potential (CEP)
Collision Cell Exit Potential (CXP)

Compound dependent optimization is performed per manufacturer's recommendation (i.e., at method development only, then determined parameters are used). Re-optimization may be performed if needed to improve or restore method performance (for example, after significant maintenance is performed, following a significant failure or event such as moving the instrument, or after the instrument is retuned or recalibrated).

Source Dependent Parameters:

Temperature

Ion Spray Voltage

Gasses

Probe position

Source dependent parameters are more susceptible to change because they are dependent on mobile phase composition and flow. Hence, these parameters are updated when mobile phase flow or composition changes.

8.3.3 Daily Check

Overall system performance is monitored daily through the injection of a perchlorate 'Shooter', which is a known concentration of perchlorate in mobile phase. The Shooter monitors target compound response from the detector, and is injected prior to any analysis. This daily check is evaluated in terms of reasonable peak retention time, shape and response. If the Shooter does not yield reasonable characteristics, problems with the system are looked for and corrected. If the Shooter characteristics are reasonable, analysis proceeds.

8.4 GENERAL SAMPLE PREPARATION

8.4.1 Every standard, sample and quality control (QC) sample must have an equivalent amount of IS added, and each sample must have similar amount of ACN/HOAc solution added in order to improve chromatography and ensure consistent peak shape in samples.

8.4.2 Standards and controls used for calibration and analytical QC such as ICAL standards, ICV, CCV, ICB, CCB are prepared as follows without filtration:

Pipette one mL of standard or control into an autosampler vial, add 10uL of WIS, 200uL of ACN/HOAc, cap tightly. Prefiltered field samples may also be prepared in this manner.

8.4.3 Soil samples, unfiltered water samples and batch preparation QC such as method blanks (MB) and Laboratory Control Sample (LCS) must be prepared with the internal standard added prior to filtering and water extraction from soils.

Samples are spiked with an equivalent amount of WIS as was added for the analytical standards above (8.4.2). For example, if 3mL of a water sample are filtered, then 30uL of WIS is added.

After samples are extracted and/or filtered, 1mL of filtrate is added to the autosampler vial with 200uL of ACN/HOAc solution added.

8.5 INITIAL CALIBRATION

Initial calibration standards are prepared with a minimum of six concentrations (see Section 6.7.2). The range of concentrations of the initial calibration is intended to define the working range of the analytical system. One of the concentrations must be at or below the analyte reporting limit (RL). In addition, a 'Shooter' (high concentration of analyte in MP), may be prepared to show system suitability, and a reagent blank may be injected without IS added in order to show that no interference or residual Perchlorate is present in the reagents used.

8.5.1 Inject 100µL of each calibration standard solution (with internal standard and organic compound mix added) into the HPLC and acquire data. Generate the calibration curve using the internal standard (isotope dilution) method.

8.5.2 A first order regression fit of six or more calibration points that is not forced through zero (e.g., least squares method) is constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be >0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit" with perfect fit being a value of 1.0. Also note that an r-value of 0.995 is equal to an r squared value of 0.99.

8.5.3 Non-linear (quadratic) regression curve fitting may be employed, with a minimum of 6 points, following the guidelines in SW-846 Method 8000. A quadratic regression may not be used to compensate for detector saturation.

8.5.4 The type of curve fit applied should be chosen to best represent the data. Strong linearity is expected for this method. If other response is observed, check for system problems. If regression criteria cannot be met, a new initial calibration (ICAL) must be performed.

8.6 INITIAL CALIBRATION VERIFICATION (ICV)

A second source (ICV) standard is analyzed immediately after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV must be within the quantitative range of the method. The acceptance criteria for the ICV are identical to those of CCV (see Section 8.8). If the acceptance criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.7 PREPARATION OF SAMPLE ALIQUOT

8.7.1 Water samples are prepared by measuring a representative volume of sample into a test tube. Sample amounts depend on sample prep and cartridge clean up and may be scaled up or down as necessary. The following example is based on a 3mL sample preparation:

- Add internal standard (30µL of 50µg/L WIS).
- Mix well and then filter with (0.45 or 0.2µm PTFE membrane or equivalent).
- Transfer 1.0mL to an autosampler vial and add 200uL of the acetonitrile/acetic acid solution.

8.7.2 Solid samples are prepared by first weighing a representative 1g solid sample aliquot into a labeled 50mL centrifuge tube. MB or LCS samples are prepared with 1g of Ottawa sand. The solid sample preparation may be scaled up (i.e., 2.0g to 20mL reagent water) if additional extract volume is needed for column and/or cartridge cleanups. The following steps are based on a 1.0g sample preparation:

- Add internal standard (100µL of 50µg/L IS spiking solution).
- Add 10.0mL reagent water (resultant IS concentration is 0.5µg/L).
- Vortex the mixture, followed by sonication for a minimum of 10 minutes, followed by additional shaking and vortex mixing.
- Visually adequate separation of solids must be achieved prior to filtering the sample. Use a centrifuge to spin the samples if appropriate (i.e., centrifuge at approximately 3200rpm for 15-25 minutes).
- Filter the supernatant extract solution using a plastic syringe fitted with a 0.45 or 0.2µm PTFE membrane filter. Dispense the filtrate into an autosampler vial for analysis.
- Cleanups may be performed per SW6850 (C_{18} cartridges for removal of organic interference) and/or EPA 331.0 (Ag, H^+, Ba^{2+} cartridges) if useful for achieving performance goals.
- Transfer 1.0mL to an autosampler vial and add 200uL of the acetonitrile/acetic acid solution.

NOTE: If sample dilution is required due to high analyte concentration or high interference, the sample may have IS added after the dilution if the dilution exceeds a factor of 10. If the dilution factor is less than 10, the sample may be

reanalyzed with the original IS.

8.7.3 Quality control samples are prepared with each extraction batch of ≤20 samples:

- Method Blank - Reagent water added (water matrix) or Ottawa sand added (solid matrices), all reagents and steps are equivalent to field sample.
- LCS - Reagent water or clean (Ottawa sand) matrix spiked at a midpoint level, prepared as is field sample.
- MS - Field sample fortified with perchlorate spike.
- Duplicate - Dup a field sample or MS

8.8 CONTINUING CALIBRATION VERIFICATION (CCV)

The CCV is used to confirm system response throughout an analytical sequence. The concentration of the CCV is at or around the midpoint of the initial calibration. Acquire a CCV at the start of each analytical sequence, after each ten injections, and at the end of each sequence. QC samples are counted as part of the number of injections, instrument blanks are not.

The percent difference (%D, drift) must be calculated for each CCV (see equation below):

$$\%D = \left[\frac{(\text{calculated concentration}) - (\text{expected concentration})}{\text{expected concentration}} \right] (100)$$

Calibration is verified when % D is $\leq$15% at midlevel, and $\leq$50% at low level (also called LODV). If any CCV does not meet acceptance criteria, analyses should be halted and corrective action taken. Reanalyze the CCV. If the CCV still fails, the instrument must be recalibrated and all samples injected since the last compliant CCV must be reanalyzed.

8.9 RT AND ISOTOPE RATIO (^{35}CL/^{37}CL) CONFIRMATION

- RT will vary due to matrix effects.
- Peak is identified by coelution of the analyte with the IS. Perchlorate identity is confirmed by evaluating the 35Cl/37Cl isotope ratio.
- Perform evaluation of the 35Cl/37Cl isotope ratio for every sample, batch QC sample and standard.
- The theoretical ratio for daughter ions m/z 83/85 is approximately 3.05.
- 83/85 peak area ratio acceptance limit - should be within $\pm$30% of the mid range calibration standard or average of all of the CCV runs of the

analytical batch if calibration was performed previously. If ratio limits are not met, inject a new aliquot of sample. If interference is suspected, dilute the sample with HPLC water if sensitivity is adequate, otherwise use cartridge cleanup. If ratio is not improved, flag sample data as suspect. Note that other criteria and corrective actions may apply, consult the applicable client LIMS program specification.

- Internal Standard Response Verification: The IS area counts must be monitored throughout the run. IS area counts must be within $\pm$50% of the average of the IS area counts of the calibration standards if calibration is performed on the same day as analysis, otherwise use the IS area counts for the first CCV of the analytical batch. If sample IS area counts do not meet the criterion, reanalyze a fresh aliquot of the sample. If the IS counts are still outside of the criterion, check the most recent CCV IS area counts. If the most recent CCV IS area counts meet the criterion, then the sample results should be considered suspect. If the most recent CCV IS area counts do not meet the criterion, corrective action must be taken to resolve the problem. Evaluate and correct as directed in the applicable LIMS program specification.

8.10 SAMPLE ANALYSIS (IDENTIFICATION, CALCULATIONS, REPORTING)
Aqueous sample extracts (with isotopically labeled perchlorate standard) are mixed with acetonitrile-acetic acid and injected for HPLC/ESI/MS/MS determination of perchlorate:

8.10.1 Confirmation is achieved via observation of coelution between perchlorate analyte ions (m/z 83 and 85) and isotopically labeled perchlorate internal standard ion (m/z 89). Relative retention time (RRT) is calculated by dividing the analyte RT by the IS RT. Acceptance limit is 1$\pm$0.02.

8.10.2 Linear regression is typically employed, quantitation of sample concentration is based on the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)\,(V_t)(DF)}{m}$$

where:

x = concentration of the analyte (ppb; µg/L or µg kg)

y = analyte instrument response (area units)

b = calculated intercept (area units)

m = calculated slope of the line (area/conc. in ppb; µg/L or µg/kg)

V_t = total volume of concentrated extract (L)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

NOTE: The calibration fit may be weighted (i.e., 1/x weighting) as described in SW8000.

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike sample (MS) and either field sample duplicate (DUP) or matrix spike duplicate (MSD). All batch QC samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specifications for additional or alternative requirements.

9.2 BLANKS

There are three types of blanks used in this analysis: Reagent Blanks (RB), Initial and Continuing Calibration Blanks (ICB/CCB), and Method Blanks (MB).

Reagent blanks are prepared with HPLC water, ACN/HOAc solution with NO WIS added.

Calibration Blanks are prepared with HPLC water, WIS and ACN/HOAc solution, but no filtration is performed.

Method Blanks (MBs) are aliquots of matrix (i.e., water or solid) that have been prepared and analyzed in the same manner as the associated field samples. HPLC or other analyte free water is used for water samples. Ottawa sand should be used in association with solid samples. MBs are analyzed to demonstrate that interferences from the analytical system, media, glassware, and reagents are under control. Concentrations of target analytes, if any, must be less than the reporting limit (RL), or as otherwise prescribed in the LIMS program specification.

9.3 LABORATORY CONTROL SAMPLE

The LCS is analyzed to measure the accuracy of the analytical system. An LCS is similar to a matrix spike analysis (below) in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample, a field sample or matrix spike sample is performed in duplicate. The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

The matrix spike is analyzed to measure matrix effects on analyte recovery. To accomplish this, a measured amount of field sample is spiked with a known amount of analyte and % Recovery is calculated as above for the LCS. See QC Table for evaluation criteria. A minimum of one MS per day and at least one per batch of 20 is required.

$$\%R = \frac{\text{(MS Sample result-Sample result)}}{\text{Spike added}} \times 100$$

9.6 METHOD DETECTION LIMIT STUDY

A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven (7) replicates for each target analyte at a concentration level near to the capabilities of the method. The MDL study is performed as needed, at minimum, annually, following the guidance of SOP 329.

10. DEVIATIONS FROM METHOD

10.1 This SOP meets the requirements of EPA SW6850.

10.2 DoD Perchlorate Handbook Rev. 1 Change 1: See Program Specification and LIMS Nickname for the detailed conformance requirements of this reference.

10.3 EPA Method 331.0: See Program Specification and LIMS Nickname for the detailed conformance requirements of this reference.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

11.1.1 The building is equipped with a safety shower, eye wash station, fire extinguisher, fire blanket, as well as a first aid kit. All lab personnel must be trained in the use and location of these items.

11.1.2 Read the MSDSs prior to preparing standards or using any solvents or reagents for the first time.

11.1.3 Wear gloves, safety glasses, and a lab coat when working with any chemical materials (e.g., standards, solvents, reagents, or samples), handling materials or equipment potentially contaminated with chemicals, or within a laboratory area.

11.1.4 Any chemicals with a Threshold Limit Value (TLV) of less than 50ppm shall be worked with in a laboratory fume hood (e.g., solvents and acids).

11.1.5 All flammable compounds must be kept away from ignition sources.

11.1.6 Any non-original containers used to hold reagents (e.g., wash bottles or automatic dispenser bottles) should be labeled at a minimum with compound name, NFPA Health, Flammability, and Reactivity ratings, and date.

11.1.7 All compressed gas cylinders must be secured at all times a regulator is in place. The cylinder cap must be installed immediately after removing the regulator and before removing the tie down strap or chain from the cylinder. The cylinder shall be secured to a gas cart for transport.

11.1.8 Food and drink are prohibited in all lab areas.

11.2 WASTE DISPOSAL

11.2.1 Mobile phase waste is collected and disposed of in accord with SOPs 003 and SOP 015.

11.2.2 All radioactive samples and sample preparations shall be disposed as per SOP 015.

11.2.3 All non-radioactive hazardous waste will be disposed of as per SOP 003.

11.2.4 All empty solvent bottles are disposed of according to the appropriate SOPs. Please note that all labels and markings must be defaced or the bottle labeled as empty prior to disposal.

12. REFERENCES

12.1 EPA Method SW6850 Rev. 0 "Perchlorate in Waters, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS/MS)". January 2007.

12.2 DoD Perchlorate Handbook; August 2007 Revision 1, Change 1.

12.3 EPA 331.0 Rev. 1.0 January, 2005 "Determination of Perchlorate in Drinking water By Liquid Chromatography Electrospray Ionization Mass Spectrometry" EPA Document # 815-R-05-007.

DOCUMENT REVISION HISTORY

12/31/08: Updated SOP Header to reflect ALS. Added paragraph to SUMMARY referencing program specification concepts. Minor text edits throughout. Added ICSS and ICSL reagents as Sections 6.8 and 6.9, respectively. Calibration and Tuning Section 8.3 was expanded. The last three bullets of Section 8.9 were edited. QC Table was updated (RT, Isotope Area Ratio, IRCS); also included program specification caveat in Table header. Added DoD program specification as Appendix A.

Analytical Method: EPA SW6850	**Parameter:** Perchlorate		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Note: The QC requirements depicted in this Table represent the laboratory's standard requirements and those of EPA SW6850. Consult applicable client program specification for QC requirements that supercede these criteria.			
Method Reporting Limit (MRL)	Demonstrated with every initial calibration	MRL ≥ lowest calibration standard	Correct system problem if calibration including MRL fails.
Limit of Quantitation (LOQ)	Demonstrated with every initial calibration	Documented in the specific matrix of concern, at or below the applicable regulatory limit Demonstrated with low calibration standard. Must be within 50% of true value	Apply J-flag to all results between the LOD and LOQ
Method Detection Limit (MDL), (LOD)	Upon implementation of method; annually and when major method changes are made; quarterly MDL checks suffice provided no significant method changes are made	MDL study must be performed in the matrix of interest using a standard at 1-10x MDL MDL must be validated through the analysis of a low-level matrix spike at approximately 2x MDL, must have SN ≥3 and isotope ratio of ± 30% of the ICAL mean	Run MDL verification at higher level and set MDL higher or re-perform MDL study. A valid MDL must be completed prior to analyzing samples.
Initial Calibration (ICAL); minimum six-point	At method set up and as needed (i.e., when daily calibration verification does not meet criteria) and after major maintenance	Minimum of 6 calibration standards (+ blank) to establish linearity, r≥0.995 (r^2 ≥0.99) The calibration is linear and shall not be forced through the origin. The concentration corresponding to the absolute value of the calibration curve's Y-intercept should be ≤ LOD. Or use mean response if the RSD for each analyte is ≤20% (including MRL)	Repeat ICAL. No samples may be analyzed and reported until calibration has been achieved and verified.
Initial Calibration Verification (ICV); second source standard at midpoint concentration	After each ICAL (conc. within cal. range)	% Difference (%D) ≤15%D relative to true value	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards. Problem must be corrected prior to analyzing and reporting samples.
Matrix Conductivity Threshold (MCT) study, performed to determine the maximum tolerable	At initial setup and when major changes occur in method operating procedures	Conduct study and determine MCT as discussed in the method.	The MCT for this analytical system using this method has been determined to be 16620uS/cm (2000mg/mL)

Analytical Method: EPA SW6850	**Parameter:** Perchlorate		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Note: The QC requirements depicted in this Table represent the laboratory's standard requirements and those of EPA SW6850. Consult applicable client program specification for QC requirements that supercede these criteria.			
concentration of known ion suppressors			
Continuing Calibration Verification (CCV)	All samples must be bracketed by the analysis of a standard demonstrating detection and accurate quantitation Analysis of mid-level standard after every 10 field samples. and at end of seq. Alternate LL and mid level concentration.	±15% ML; ± 50% LL (LL = approx. RL) Note: LL CCV equiv. to LODV	Evaluate/correct instrument malfunction as needed (e.g., rinse column, lines); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. Samples analyzed after a failed CCV must be reanalyzed. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed.
Relative Retention Time (RRT)	Evaluated with each acquisition	The RRT between m/z 83 and m/z 89 must be less than 1.0± 2% (0.98-1.02)	Determine cause of problem and take corrective action
Isotope Area Ratio (m/z 83/85) to confirm presence of perchlorate	Evaluated with each acquisition (for every standard, sample, and control)	83/85 peak area ratio must fall between ±30% of ratio of the mid range calibration standard or the average ratio of all CCVs of the analytical batch if calibration was performed on a previous day	If ratio limits are not met, inject a new aliquot of sample. If interference is suspected, dilute if sensitivity is adequate or use cartridge cleanup. If ratio is not improved, flag sample data and narrate. (Note: Laboratory practice; corrective action not specified in SW6850)
Internal Standard Response Verification (IS = IRCS)	Same level added to every standard sample or control.	IS area counts must be within ±50% of the average of the IS area counts of the calibration standards if calibration is on the same day as analysis, otherwise use the IS area counts for the 1st CCV of the analytical batch	If IS area counts do not meet acceptance criterion, analyze a second aliquot. If the IS counts are still outside of the criteria, check the most recent CCV IS area counts. If the most recent CCV IS area counts meet the criteria, then the sample results should be considered suspect. If the most recent CCV IS area counts do not meet the criteria, corrective action must be taken to

Analytical Method: EPA SW6850	**Parameter:** Perchlorate		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Note: The QC requirements depicted in this Table represent the laboratory's standard requirements and those of EPA SW6850. Consult applicable client program specification for QC requirements that supercede these criteria.			
			resolve the problem.
ICB and CCB (Note: not required SW6850)	Immediately following the corresponding calibration verification samples	<RL, or as otherwise specified in the applicable LIMS program specification	Correct problem, demonstrate freedom from blank interferences and quantitative performance.
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix	Perchlorate concentration <RL, or as otherwise specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition.
Laboratory Control Sample (LCS)	1 per preparation batch of ≤20 samples of like matrix, spiked at the RL.	Recovery within 80-120%, or as otherwise specified in the applicable LIMS program specification	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may need to be reanalyzed) - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration

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Analytical Method: EPA SW6850	**Parameter:** Perchlorate		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Note: The QC requirements depicted in this Table represent the laboratory's standard requirements and those of EPA SW6850. Consult applicable client program specification for QC requirements that supercede these criteria.			
Matrix Spike (MS)	1 per preparation batch of ≤20 samples of like matrix	Recovery within MS limits: 80-120%. Liquid; 70-130%. Solid, or as otherwise specified in the applicable LIMS program specification	Evaluate data to determine the source of the recovery problem or difference in replicates.
Matrix Spike Duplicate (MSD) or Field Sample Duplicate (DUP)	One MSD or sample Dup. per batch	RPD < 15% when at or above mid range of the ICAL; RPD < 50% when at low end of the ICAL	Examine data, check IS performance and integration. Re-prep. if appropriate.

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ALS Laboratory Group - Fort Collins

Perchlorate 6850 Bench Prep Sheet

Shooter __ul Inter A 1st Source in ____mL MP - System Check OK ___ Date _____ Operator _____ WO # (s)_____ Nickname(s) _____

Instrument ID **LC/MS/MS** SOP ____ / Rev ___ Matrix _____ Filter lot_____ Cartridge lot(s) (if applicable) _____

D:Analyst Data\Projects\Perchlorate\... Acquisition Method:..PercMRM___112808___.divert Batch Name: ...Batch_____.dab \Data Path: ...Data_____.wiff

Quantitation Method:.. _____.qmf Results table:._____.rdb ICAL Result File: _____ ICAL mean IS Area _____ ICAL Isotope Ratio _____

Stock Int. A ST080208-5 **Int. B** ST080208-6 **LODV** ST080212-2 **CCV**(m) ST080212-4 **CCV**(h) ST080212-7 **ICSL** ST080212-5 **LCS**(rl) ST080212-3 **LCS**(m) ST080212-6 **LCS**(h) ST080212-8 **Exp.** 2/1/2009

WIS ST080212-9 exp.9/21/2008; **ICV** ST071202-22 exp.8/6/2008; **ACN/HOAc** RG080213-1 **HPLC Water** 072343 **Others**_____

#	Sample Name	Method / Nickname	Sample Head Space? **Y** or **N**	Conductivity (µS/cm) (if done)	Matrix	Starting Matrix / Amt.	Extract H_2O Added (mL)	ID Stock Added	Amount Stock Added (varies)	WIS Added (µL)	Filtered? **Y** or **N**	Amount Prep. Added to AS Vial (mL)	ACN/ HOAc Added (mL)	Vial Position	Comments
						Example - use most current iteration									

Reviewed by / date _____

Form 415r1.xls (3/27/08)

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APPENDIX A (INTERNAL USE ONLY)
DoD Program Specification

(per DoD Perchlorate Handbook, Rev1, Change1)

QC Check	Frequency	Acceptance Criteria	Summary of Quality Control (QC) Procedures and Corrective Actions
Reagent Blank	Prior to calibration, after over-range samples; after batch analysis	<½ RL	Reanalyze reagent blank and determine source of problem. If reagent contamination is determined, replace source and reanalyze. If carryover is determined, correct problem and reanalyze. Apply B-flag to all samples that cannot be reanalyzed.
ICAL	At method setup and after major maintenance	RSD <20% ea. Linear Fit-No Force Conc. = [Y-inter.] ≤ LOD r= > 0.995; ≥ 5 stds	Correct problem and repeat ICAL. No sample may be run until ICAL has passed.
ICV (2nd Source)	After every ICAL	±15% (mid-range conc.)	Correct problem and re-run. No samples may be run until ICV has passed.
MCT	At initial setup and when major changes occur in method operating procedures	Conduct study and determine MCT	The MCT for this analytical system by this method has been determined to be 16620uS/cm (2000mg/mL)
ICS (ICSL)	1x/20 samp. Batch	Same pretreatment as samples Conc. @ RL [Anion] at MCT Result ±30%	Correct problem then reanalyze batch. No samples may be reported with a failing ICSL.
CCV	After every 10 samples	Rec.±15% (mid-range conc.)	Correct and re-run all samples since the last acceptable CCV. If that fails, or samples cannot be re-run, Q flag all affected samples.
MB	At least one per batch of 20 samples < ½ RL	MB conc. < ½ RL	Correct problem, re-prep and reanalyze MB and all samples associated with the batch. Apply a B-flag to all results if the samples cannot be successfully reanalyzed.
LCS	1x/20 @RL	Rec.±20%, or within laboratory-generated limits, whichever is more stringent	Correct the problem then re-prep and re-analyze LCS and all associated samples. If that fails, or samples cannot be re-run, Q flag all affected samples.
LODV	Bracket Batch of 20 samples. @ 2x LOD	Rec. ±30% LODV Failure: Samples in the batch at > LOD < RL must be re-evaluated. Samples above the RL may be reported.	Correct problem and rerun the LODV and all samples since the last failing LODV (as described under acceptance criteria). Apply a Q flag to all samples since the last unacceptable Q-flag if reanalysis is not possible.
MS	≥1per prep. Batch of 20 samples; @RL	Rec.±20%, or within laboratory-generated limits,	In parent sample apply J flag if acceptance criteria are not met.

APPENDIX A (INTERNAL USE ONLY)
DoD Program Specification

(per DoD Perchlorate Handbook, Rev1, Change1)

QC Check	Frequency	Acceptance Criteria	Summary of Quality Control (QC) Procedures and Corrective Actions
		whichever is more stringent	
Laboratory Dup. or MSD	≥1per prep. Batch of 20 samples spiked at RL.	RPD <15%	In parent sample apply J flag if criteria not met.
IS Recovery (IS = IRCS)	Same level added to every standard sample or control.	±50% of the average IS area counts of original ICAL	Determine cause of problem and take corrective action. If interference is suspected dilute and reanalyze. If correction is not possible Q flag and narrate.
Relative Retention Time	Evaluated with each acquisition	The RRT between m/z 83 and m/z 89must be less than 1.0±2% (0.98-1.02)	Determine cause of problem and take corrective action. If interference is suspected dilute and re-analyze. If correction is not possible Q flag and narrate.
Isotope Area Ratio (m/z 83/85)	Evaluated with each acquisition (for every standard, sample, and control)	Theoretical 3.06; must fall 2.3-3.8 (25% window)	If ratio limits are not met, inject a new aliquot of sample. If interference is suspected, dilute if sensitivity is adequate or use cartridge cleanup. If ratio is not improved, flag sample data and narrate.

Note that a valid MDL study and check per ALS-FC standard practice are required for DoD analyses.

CT Laboratories
Baraboo Laboratory Division

Title: **Semi-Volatile Organochlorine Pesticide Analysis**

SOP Number: **8081**

Written by: _____

Technical Review by: _____

Approved by: _____
Quality Assurance Manager

Approved by: _____
Laboratory Director

SOP Manual Control Number: _____

1.0 SCOPE/APPLICATION

1.1 Method SW-846 8081 is used to determine the quantitation of various Organochlorine Pesticides in extracts from solid and aqueous matrices. Capillary columns are employed with electron capture detectors (ECD). Target compounds determined by this method are listed in Table 1.0.

1.2 This method is a dual column analysis. Each column uses the same QC criteria for the reporting of analytical results. The analytical columns are joined to a single injection port. The secondary chromatographic column is used to confirm or report final results in conjunction with the primary column.

1.3 Toxaphene and tech-Chlordane are multi-component mixtures. The identification of the multi-component analytes can be performed by a combination of pattern recognition and retention time. Quantitation is based upon indicator peaks that are generally present in each compound. Three to five peaks are chosen for each compound and each peak is then calibrated to represent that compound, resulting in separate calibration methods for Toxaphene and tech-Chlordane.

1.4 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

Table 1.0
Pesticides with Method Detection Limits and Limits of Quantitation

Analytes	Water MDLs (ug/L)			Soil MDLs (mg/kg)	
	MDLs	LOQs		MDLs	LOQs
4,4'-DDD	0.006	0.020		0.0003	0.0012
4,4'-DDE	0.006	0.020		0.0003	0.0011
4,4'-DDT	0.007	0.022		0.0005	0.0015
Aldrin	0.006	0.017		0.0005	0.0017
alpha-BHC	0.005	0.016		0.0006	0.0019
alpha-Chlordane	0.009	0.030		0.0003	0.0011
Beta-BHC	0.009	0.030		0.0006	0.0020
Chlordane (tech)	0.102	0.325		0.0040	0.0150
Delta-BHC	0.005	0.017		0.0003	0.0011
Dieldrin	0.006	0.018		0.0003	0.0012
Endosulfan II	0.007	0.022		0.0003	0.0012
Endosulfan sulfate	0.006	0.020		0.0009	0.0031
Endrin	0.006	0.020		0.0004	0.0014
Endrin aldehyde	0.009	0.028		0.0011	0.0036
Endrin ketone	0.007	0.021		0.0008	0.0028
Gamma-Chlordane	0.007	0.024		0.0003	0.0011
Heptachlor	0.006	0.019		0.0004	0.0012
Heptachlor epoxide	0.007	0.021		0.0005	0.0017
Lindane	0.007	0.022		0.0005	0.0016
Methoxychlor	0.006	0.020		0.0007	0.0023
Mirex	0.010	0.020		0.0010	0.0020
Toxaphene	0.176	0.562		0.0050	0.0290

Table 1.1

Pesticides and Retention Times

Pesticide Name	Retention Time
alpha-BHC	1.96
Lindane (gamma-BHC)	2.47
beta-BHC	2.59
delta-BHC	3.04
Heptachlor	3.59
Aldrin	4.16
Heptachlor Epoxide (b)	4.95
gamma-Chlordane	5.42
Endosulfan I	5.60
alpha-Chlordane	5.73
Dieldrin	6.20
4,4'-DDE	6.37
Endrin	6.69
Endosulfan II	7.08
4,4'-DDD	7.57
Endrin Aldehyde	7.72
Endosulfan Sulfate	8.47
4,4'-DDT	8.60
Endrin Ketone	9.59
Methoxychlor	10.26
Mirex	10.50

Table 1.2
Toxaphene and tech-Chlordane Retention Times

Compound Name	RT Peak#1	RT Peak#2	RT Peak#3	RT Peak#4	RT Peak#5
tech-Chlordane	3.25	3.58	5.41	5.72	5.84
Toxahene	6.69	7.52	8.48	8.80	9.23

Note: Retention time shifts can occur when instrument maintenance is performed. The shifts in the retention time are reflected in the analytical method.

Table 1.3

Laboratory Control Sample Limits

	Inhouse					QSM			
	Water (ug/L)		Soil (mg/kg)			Water (ug/L)		Soil (ug/kg)	
Analyt	Lower Limit	Upper Limit	Lower Limit	Upper Limit		Lower Limit	Upper Limit	Lower Limit	Upper Limit
alpha-BHC	57	123	53	120		60	130	60	125
Lindane	66	119	55	122		25	135	60	125
beta-BHC	61	128	51	125		65	125	60	125
delta-BHC	33	118	36	117		45	135	55	130
Heptachlor	54	122	58	121		40	130	50	140
Aldrin	55	124	56	123		25	140	45	140
Heptachlor Epoxide (b)	70	123	57	122		60	130	65	130
gamma-Chlordane	67	124	56	121		60	125	65	125
Endosulfan I	66	128	57	122		50	110	15	135
alpha-Chlordane	69	123	55	123		65	125	65	120
Dieldrin	69	126	55	124		60	130	65	125
4,4'-DDE	69	125	55	125		35	140	70	125
Endrin	57	147	46	143		55	135	60	135
Endosulfan II	65	132	57	123		30	130	35	140
4,4'-DDD	64	130	55	128		25	150	30	135
Endrin Aldehyde	59	126	32	120		55	135	35	145
Endosulfan Sulfate	49	131	50	121		55	135	60	135
4,4'-DDT	65	128	54	126		45	140	45	140
Endrin Ketone	63	134	58	124		75	125	65	135
Methoxychlor	63	132	53	131		55	150	55	145
t-Chlordane									
Toxaphene									
TCMX	43	120	59	124		25	140	70	125
DCB	39	143	48	136		30	135	55	130

Note: Information on recovery limits and RPDs are generated though StarLIMS. The information tables are stored in H:\Quality Systems\QC\Charting.

Table 1.4

Matrix Spike/Matrix Spike Duplicate Limits

	Inhouse						QSM					
	Water (ug/L)			Soil (mg/kg)			Water (ug/L)			Soil (ug/kg)		
Analyt	Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit	RPD	Lower Limit	Upper Limit	RPD
alpha-BHC	62	119	26	20	148	34	60	130	30	60	125	30
Lindane	63	121	36	30	133	33	25	135	30	60	125	30
beta-BHC	58	131	34	32	139	30	65	125	30	60	125	30
delta-BHC	26	122	26	30	124	44	45	135	30	55	130	30
Heptachlor	64	126	28	27	159	32	40	130	30	50	140	30
Aldrin	63	120	28	38	141	28	25	140	30	45	140	30
Heptachlor Epoxide (b)	66	128	32	43	134	38	60	130	30	65	130	30
gamma-Chlordane	56	137	29	36	140	35	60	125	30	65	125	30
Endosulfan I	61	137	41	51	124	29	50	110	30	15	135	30
alpha-Chlordane	62	129	28	42	128	34	65	125	30	65	120	30
Dieldrin	56	138	35	40	135	37	60	130	30	65	125	30
4,4'-DDE	58	132	32	45	147	22	35	140	30	70	125	30
Endrin	51	159	27	30	169	39	55	135	30	60	135	30
Endosulfan II	63	134	28	54	125	36	30	130	30	35	140	30
4,4'-DDD	30	132	44	43	136	24	25	150	30	30	135	30
Endrin Aldehyde	59	129	44	24	132	40	55	135	30	35	145	30
Endosulfan Sulfate	43	136	38	33	143	41	55	135	30	60	135	30
4,4'-DDT	56	140	36	46	141	35	45	140	30	45	140	30
Endrin Ketone	63	136	32	36	143	25	75	125	30	65	135	30
Methoxychlor	54	140	34	50	148	35	55	150	30	55	145	30
t-Chlordane												
Toxaphene												
TCMX	60	121		39	138		25	140		70	125	
DCB	38	144		36	151		30	135		55	130	

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous and soil matrices (reference methods SW846-3510 and 3545), concentration techniques that are suitable for preparing the extract, and the quantitative/qualitative analysis for the determination of pesticides by method SW846-8081.

2.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method applies for aqueous samples extracted by liquid-liquid separatory funnel (SW846-3510). Method applies for soil/sediment, and solid waste samples extracted by standard solvent extraction methods utilizing pressurized extraction techniques as heated pressurized fluid

extraction (SW846-3545). This method includes the extraction for waste oil samples.

2.3 The resultant extract is chemically dried and concentrated in a Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

2.4 Extracts for pesticide analysis can be subjected to a variety of cleanup steps, depending on the nature of the matrix interferences and target analytes. Suggested cleanup methods (see attachments I, II, and III) include; Florisil (Method 3620), sulfur cleanup (Method 3660), and Gel-Permeation Chromatography (GPC) cleanup (Method 3640A). Alternative cleanup methods (refer to SW-846) are; alumina (Method 3610) and silica gel (Method 3630). After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with dual capillary columns and ECD detectors.

2.5 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

3.0 DEFINITIONS

3.1 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.2 Laboratory Control Spike (LCS): Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike is used to document the ability of an analyst to generate acceptable precision and bias, to verify the analytical system's performance, and to document method accuracy for each matrix.

3.3 Laboratory Control Spike Duplicate (LCSD): A duplicate Milli-Q water (for water) and Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike duplicate is used to document the precision and bias of a method. This default QC type is only performed when insufficient amount of sample is available for performing a matrix spike and matrix spike duplicate. Toxaphene and t-Chlordane are not spiked into the LCSD.

3.4 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias

of a method in a given sample matrix. Toxaphene and t-Chlordane are not spiked into the MS.

3.5 Matrix Spike Duplicate (MSD): Intra-laboratory split samples spiked with identical concentration of target analytes. The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix. Toxaphene and t-Chlordane are not spiked into the MSD.

3.6 Method Reporting Limit (MRL): The method reporting limit is a threshold value below which the laboratory reports a result as non-detect. The highest value reported for the method reporting limit is dependant upon project-specific action or decision levels. Method reporting limits are adjusted based on the sample matrix and any sample dilution/concentrations when necessary.

3.7 Method Detection Limit (MDL): The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. It is determined from analysis of a sample in a given matrix containing the analyte. Depending on project specifics, MDLs will be in the form of a MDL check standard or the MDL is subject to extraction procedures. An acceptable MDL standard check must produce a signal at least 3 times the instrument's noise level. Also, a MDL check injection must be made after each major instrument repair or changing of instruments (prior to sample analysis) to determine the performance sensitivity of the analysis.

3.8 Surrogate (SURR) An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Its use is to monitor the performance of the extraction, cleanup (as needed), the analytical system, and the effectiveness of the method. The surrogates are; 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl.

3.9 Initial Calibration (ICAL): An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of an analyte. This relationship is depicted graphically, and referred as to a "calibration curve". Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte.

3.10 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed.

3.11 Continuing Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis.

3.12 Evaluation Verification Standard (EVAL): This standard solution, containing Endrin and DDT, uses the area counts of these compounds and their breakdown products to assess instrument conditions.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems can be necessary. Refer to each method for specific guidance on quality control procedures.

4.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.3 Soap residue (e.g. sodium dodecyl sufate), which results in a basic pH on glassware surfaces, can cause degradation of certain analytes.

4.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample will be necessary.

4.5 Elemental sulfur (S_8) is readily extracted from soil samples. High concentrations of sulfur will cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through cleanup procedures such as Method 3660. Since the recovery of Endrin aldehyde is drastically reduced when using the TBA procedure in Method 3660, this compound must be determined prior to sulfur cleanup when it is an analyte of interest and the TBA procedure is to be used for cleanup.

4.6 Other non-target halogenated pesticides or industrial chemicals can interfere with the analysis of pesticides. Co-eluting chlorophenols will be eliminated by florisil treatments. Aroclors also interfere with analysis of organochlorine pesticides. The biggest challenge is for the multi-component analytes such as tech-Chlordane and Toxaphene. If Aroclors are expected to occur in samples, the analyst shall consult Methods 3620 (florisil) and 3630 (silica gel) for techniques that can be used to separate the pesticides from the Aroclors.

5.0 SAFETY

5.1 Protective clothing; safety glasses, gloves, apron and/or lab coat, long pants, and protective shoes, will be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure will utilize appropriate laboratory safety systems.

5.2 The toxicity of chemicals used in this method has not been precisely defined. Each chemical will be treated as a potential health hazard, and exposure to these chemicals will be minimized.

6.0 APPARATUS & MATERIALS

Apparatus:

6.1 **Gas Chromatograph**- An analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including syringes, analytical columns, electron capture detectors, auto sampler, electronic pressure control, and EZ Chrom Elite data acquiring system.

6.1.1 Carrier Gas: Hydrogen at 37.0 psi at 18.7 ml/min.
Mode: Constant Pressure
Injection Volume: 1 ul
Injector: 250 ^{o}C with Supelco Septa SS-174
Mode: Pulse Splitless
Pulse Pressure: 50 psi
Pulse Time: 0.65 min.
Purge Time: 0.48 min.
Purge Flow: 10 ml/min.

Detector (s): 295 ^{o}C
Mode: Constant Makeup
Make-up Gas: Nitrogen at 35 mL/min.
Range: 4 on A and B channels

Oven: Initial – 150 ^{o}C (hold for 1.65 minute)
Ramp – 10 ^{o}C/minute
Final - 185 ^{o}C (hold for 0.5 minutes)
Ramp – 3 ^{o}C/minute
Final - 190 ^{o}C (hold for 0.5 minutes)
Ramp – 11°C/minute
Final - 200 ^{o}C (hold for 0.50 minutes)
Ramp – 18.50 ^{o}C/minute

Final - 280 °C (hold for 0 minutes)

Note: Instrument operating parameters are subject to change to improve overall chromatography (changes are noted in the Instrument Maintenance Log Book).

6.2 **Analytical column pair**;

6.2.1 30m x 0.32 mm ID bonded with 5% phenyl polysiloxane / 95% dimethylsiloxane, 0.25 um. (ZB-5, part # 7HM-G002-11 or equivalent).

6.2.2 30m x 0.32 mm ID bonded with 14% Cyanopropylphenyl/86% dimethylplysiloxane, 0.25 um (ZB-1701, part # 7HM-G006-11 or equivalent).

6.3 Water bath- heated and capable of accepting a Kuderna-Danish apparatus.

6.4 Dionex ASE 200.

6.5 Organomation Nitrogen blow down concentrator.

6.6 Analytical balance capable of accurately weighing to the nearest 0.01 gram (for sample preparation).

6.7 Oven.

Glassware:

6.8 Separatory funnel - 2000 mL glass with Teflon stopcock.

6.9 Kuderna-Danish (K-D) apparatus:
- 6.9.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).
- 6.9.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).
- 6.9.3 Synder column- Three-ball macro (Fisher # K503000-0121).
- 6.9.4 Springs or Teflon clamps to attach concentrator tube to evaporation flask.

6.10 Graduated cylinder (Class A) - 1000 mL. (Fisher 08-559G).

6.11 Beaker - 250 mL and 600 mL.

6.12 Vials - 2.0, 12, and 60 mL screw cap vials with Teflon lined caps (Fisher # 03-377-10B).

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6.13 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

6.14 Funnels – glass.

6.15 Volumetric flask – (Class A) 10, 25, 50, and 100 mL.

6.16 Syringes 10 uL, 100 uL, 500 uL, and 1,000 uL.

Reagents:

6.17 Deionized water (Milli-Q processed), analyte free.

6.18 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212). Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil.

6.19 Silica sand- hydrocarbon free. Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil.

6.20 Methylene chloride, pesticide grade, analyte free.

6.21 Acetone, pesticide grade, analyte free.

6.22 Hexane, pesticide grade, analyte free.

6.23 Isopropanol, pesticide grade, analyte free.

6.24 Hydrogen (99.995% purity or greater).

6.25 Nitrogen (99.995% purity or greater).

6.26 Sulfuric Acid-1:1(v/v). ACS grade. Store in lab at room temperature.

6.27 Sodium Hydroxide- 10 N. ACS grade. Store in lab at room temperature.

6.28 Diatomaceous earth (JT Baker # 1939-01).

Materials:

6.29 Boiling chips, carborundum, approximately 10/40 mesh (Methylene chloride rinsed). (G. Frederick Smith Chemical Company, Catalog #198)

6.30 pH indicator paper- pH 0-14. (Whatman Catalog #2613991)

6.31 Dionex ASE Filters (Catalog #049458).

6.32 Filter- Glass Microfibre 12.5 cm GF/A, (Whatman # 1827-125).

6.33 Spatulas- stainless steel.

7.0 STANDARDS AND SPIKES

7.1 Preparation of standards is documented in the Pest/PCB standards logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards once opened, expire within six months or sooner if routine QC indicates a problem, and are not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire in six months and are not to exceed the opened date of the stock standard or the manufactures expiration date.

7.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standards shall have the manufactures suggested expiration date. Stock standards, once opened, expire within six months and are not to exceed the manufactures expiration date. The following list of stock standards are certified, commercially prepared standards, such as:

Organochlorine Pesticide Mix AB#1: Restek Part # 32291 at 200 ug/mL.
Toxaphene Pesticide Mix: Restek Part # 32005 at 1000 ug/ml.
tech-Chlordane Pesticide Mix: Restek Part # 32021 at 1000 ug/ml.
Pesticide Surrogate Mix: Restek Part # 32000 at 200 ug/ml.
Pesticide System Evaluation Mix: Supelco Part # 48282 at 500 ug/ml.

7.3 Intermediate Working Stock Standards: These standards are diluted stock standards so that the concentration levels are manageable for the working range of the gas chromatograph. The pesticides standards are prepared at an optimum level from the working stock standard. Each pesticide compound is at a concentration of 2.0 ug/ml with surrogate concentration at 1.25 ug/ml in hexane (Table 2.0). For the Toxaphene and tech-Chlordane, the multi-component compounds are at a concentration of 10.0 ug/ml with a surrogate concentration of 1.25 ug/ml (Table 2.1).

Table 2.0
Intermediate Working Stock Standard Concentration

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Pesticide Mix AB	200	0.100	10	2.0
Surrogate	200	0.0625	10	1.25

*Note: The concentration of the Intermediate Working standard and Intermediate ICV standard are prepared the same manner.

Table 2.1
Intermediate Standard Concentration

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Toxaphene	1000	0.100	10	10.0
tech-Chlordane	1000	0.100	10	10.0
Surrogate	200	0.030	10	0.60

7.4 Calibration standards: An initial calibration of the listed pesticides in Table 1.0 is performed using a minimum of 5 points. Such standards will be used to demonstrate the linearity of the detectors corresponding to the expected concentrations found in real samples. This is achieved by taking known aliquots of the intermediate standard and diluting them to volume in hexane. The following levels are repeated across all pesticide compounds. Alternative standard concentrations for the calibration curve can be prepared to meet client or program specified criteria:

Linearity Points for Organochlorine Pesticides:

Level 1	0.001 ug/ml
Level 2	0.002 ug/ml
Level 3	0.008 ug/ml
Level 4	0.020 ug/ml
Level 5	0.040 ug/ml
Level 6	0.050 ug/ml
Level 7	0.070 ug/ml

Table 2.2
Calibration Points for the Pesticide Linearity

Linearity Points	Spike Concentration (ug/ml) Pesticides	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml) Pesticides	Final Concentration (ug/ml) Surrogate**
1	2.0	0.005	10.0	0.001	0.0006
2	2.0	0.010	10.0	0.002	0.0013
3	2.0	0.040	10.0	0.008	0.0050
4	2.0	0.100	10.0	0.020	0.0125
5	2.0	0.200	10.0	0.040	0.0250
6	2.0	0.250	10.0	0.050	0.0313
7	2.0	0.350	10.0	0.070	0.0438

7.5 Calibration standards: An initial calibration of the listed multi-component pesticides in Table 1.1 is performed using a single point CF or a minimum of a five point calibration curve. These concentrations correspond to the expected concentration found in real samples. Toxaphene and tech-Chlordane is be quantitated on the basis of 4 to 6 major peaks. The

analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. It is highly unlikely that the peaks will match exactly, but the analyst can not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard.

Toxaphene and tech-Chlordane:

Level 1	0.030 ug/ml
Level 2	0.050 ug/ml
Level 3	0.100 ug/ml
Level 4	0.250 ug/ml
Level 5	0.400 ug/ml
Level 6	0.600 ug/ml

Table 2.3
Calibration Points for the Toxaphene and tech-Chlordane Linearity

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)	Final Concentration (ug/ml) Surrogate**
1	10.0	0.030	10.0	0.030	0.0018
2	10.0	0.050	10.0	0.050	0.003
3	10.0	0.100	10.0	0.100	0.006
4	10.0	0.250	10.0	0.250	0.015
5	10.0	0.400	10.0	0.400	0.024
6	10.0	0.600	10.0	0.600	0.036

7.6 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target pesticides at 0.040 ug/ml and Toxaphene and tech-Chlordane at 0.25 ug/ml each time the initial calibration is performed. This is achieved by taking known aliquots of the ICV intermediate standard and diluting them to volume in hexane.

Table 2.4
ICV Working Pesticide Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Pesticides	2.0	0.200	10	0.040
Surrogate	1.25	0.200	10	0.025

Table 2.5
ICV Working Toxaphene and tech-Chlordane Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Tox / Chlor	10.0	0.250	10	0.250
Surrogate	0.60	0.250	10	0.015

7.7 Continuing Calibration Verification Standard (CCV): A working standard solution for organochlorine pesticides at a concentration of 0.040 ug/ml and Toxaphene and tech-Chlordane at 0.25ug/ml is used to check the validity of a calibration curve on a daily basis. This is achieved by taking known aliquots of the intermediate standard and diluting them to volume in hexane.

Table 2.6
CCV Working Pesticide Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Pesticides	2.0	0.200	10	0.040
Surrogate	1.25	0.200	10	0.025

Table 2.7
CCV Working Toxaphene and tech-Chlordane Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Tox / Chlor	10.0	0.250	10	0.250
Surrogate	0.60	0.250	10	0.015

7.8 Surrogate standard: A commercially prepared certified solution of 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl is diluted in acetone to produce a working surrogate solution of 0.50 ug/mL. 0.5 mL is added to each sample and QC.

Table 2.8

Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Surrogate	200	0.250	100	0.500

7.9 Spiking standards (matrix and control samples): Prepare a spiking solution in acetone that contains pesticides and concentration of 0.80 ug/ml and Toxaphene and tech-Chlordane at 10.0 ug/mL for water and sediment / soil samples. 0.50 mL is added to quality control and matrix spike samples. The concentration of these compounds can be five times higher for waste samples. If other compounds of interest are to be monitored, they can be added at an appropriate level and noted in the standard preparation log. If client request a specified pesticide, a spiking solution will be altered to match the pesticide of interest.

Table 2.9
Pesticide Spiking Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Pesticides	200	0.200	50.0	0.800

Table 2.10
Toxaphene and tech-Chlordane Spiking Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Toxaphene	1000.0	0.50	50.0	10.0
t-Chlordane	1000.0	0.50	50.0	10.0

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. Soil samples must be extracted within 14 days and water samples must be extracted within 7 days from the date of collection.

8.2 Sample extracts are stored under refrigeration in the dark and analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

9.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in µg/L and the relative standard deviation (RSD) of the recoveries for each analyte. In addition to each set of IDCs, a blind laboratory spike will be performed. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000, sec. 9.4).

9.3 Many programs (i.e. LCG, QSM) require the analysis of method reporting limit (MRL) standards and method detection limit (MDL) check standards as another means of checking method performance. MRLs are analyzed at the beginning and end of each 12 hour shift, and are typically prepared at concentrations equal to the reporting limits of the program. Recovery limits are program specific, but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x to 3x the method detection limit. MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). MDL check standards are derived from diluting the stock standard to a known concentration of 2x or 3x the method detection limit (Louisville Chemistry Guidelines, LCG). In most instances, a method detection limit check sample is analyzed at the beginning and end of each sequence requiring MRL standards. If the MRLs fail for any compound, a MDL must be analyzed immediately following the failed MRLs. The recovery criteria for MDL check samples is the ability to detect all compounds at three times the noise level. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

9.4 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SURR, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for

generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit of 20 % RSD are used until enough data points are generated to provide usable internal limits. Programs, client and/or project specific limits are used frequently in sample analyses. Refer to the QSM, DOD, ACOE, LCG, or client QAPPs for program specified limits. In the case where no specified limits are supplied or referred to, in-house limits will be used. The Quality Control Requirements chart (Table 3) also lists specific criteria to the method/project/program.

10.0 CALIBRATION

10.1 Calibration

10.1.1 The initial calibration for SW-846-8081 chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument across two dissimilar analytical columns. Each column must meet the initial calibration and continuing calibration criteria found in Table 3.

10.1.2 The identification of the analytes, such as pesticides Toxaphene and t-Chlordane, can be performed by a combination of pattern recognition and retention time. Retention time windows are crucial to the identification of target compounds. Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in accordance with Method 8000 Section 11.6. Make three injections of target analyte standards over a course of 72-hour period. Each retention time must be to three decimal places. Calculate the mean and standard deviation of the target analytes. The width is 3x the standard deviation of the mean absolute retention time during the 72 hour period or 0.03 minutes, which ever is higher. If the standard deviation is 0.000, the laboratory will include more data points or use the default value of 0.01 minutes. Each analyte in each standard must fall within its respective retention time window. The retention time shall be set using the midpoint standard of the initial calibration curve or the value in the continuing calibration verification standard at the beginning of the analytical shift. If the analytes fall outside the established retention time window, the gas chromatographic system must either be adjusted so that a second analysis of the standard results in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.1.3 An initial calibration of the listed multi-component pesticides in Table 1.1 is performed using a single point CF or a minimum of a five point calibration curve. These concentrations correspond to the expected range found in real samples. Quantitation is based

upon indicator peaks that are generally present in these multi-component mixtures. Three to five peaks are chosen for each compound and each peak is then calibrated to represent that compound, resulting in separate calibration methods for Toxaphene and tech-Chlordane. Each peak actually has a ratio concentration of the final concentration of that analyte, but when the method sums that concentration, it generates the correct final concentration.

10.1.4 For each compound and surrogate of interest, calibration standards are prepared at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. Note: QSM requires that the LOQ or the lowest point in the curve, which is greater, be used for the reporting limit.

10.1.5 The lowest concentration calibration standard that is analyzed during an initial calibration curve establishes the method's quantitation limit based on the finale volume of the sample extract described in the preparative method or employed by the laboratory.

10.1.6 External standard calibration involves comparison of instrument response from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount of analyte in the calibration standard is defined as the calibration factor (CF). Toxaphene and tech-Chlordane are multi-component standards. Three to five peaks will be selected from each and calibrated using the average of the peaks.

10.1.7 Linear calibration using the average calibration or response factor. When calculating, both calibration factors and response factors are a measure of the slope of the calibration relationship and assume that the curve passed through the origin. If the relative standard deviation (RSD) is less than or equal to 20%, the use of the linear model is generally appropriate, and the calibration curve can be assumed to be linear and to pass through the origin

10.1.8 Linear calibration using a least squared regression. When the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The approach is to employ a regression equation that does not pass through the origin. This approach can also be employed based on past experience of the instrument response.

10.1.9 Non-linear calibration. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable, and monotonic over the calibration range. The

statistical considerations in using a non-linear calibration model require more data than the traditional linear approaches. A quadratic (second order) model requires the use of six initial calibration points.

10.1.10 Directly inject the prepared calibration standards into the gas chromatograph. An external calibration technique is employed. One of the external standards will be at a concentration near, but above the method detection limit. If the RSD $\leq$ 20%, the correlation of $r \geq 0.995$, or $r^2 \geq 0.990$ is obtained then the calibration is deemed acceptable.

10.1.11 Check the validity of the calibration by analyzing an ICV. The variance of any given compound shall not be more than +/- 15% difference. This standard must be run prior to sample analysis. If the percent drift or the percent difference criteria is not met, then the subsequent sample analysis for that analyte is not acceptable.

10.1.12 Continuous checking of the validity of the calibration by analyzing CCVs. The variance of any given compound shall not be more than +/- 15% difference. This standard must be ran at the beginning, every 20 samples or every 12-hour interval (which ever comes first), and at the end of the analysis run. Toxaphene and tech-Chlordane CCV can be analyzed at the beginning or at the end of the analytical run. Note: QSM samples must have CCVs injected every ten samples for pesticides, Toxaphene, and tech-Chlordane.

10.1.13 It is highly recommended to employ two standards (CCVs) at different concentrations to verify the calibration curve using non-linear calibration. One standard shall be near the quantitation limit or action limit. The choice of specific standards and concentrations is generally a method or project specific consideration.

10.2 Calibration Calculations

10.2.1 When determining Organochlorine Pesticides, Toxaphene and tech-Chlordane by the external standard technique, calculate the calibration factor (CF) for each peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in Nanograms)}}$$

10.2.2 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation, and the relative standard deviation (RSD) for each multi-component peak.

$$\text{Mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (CF_I - \overline{CF})^2}{n-1}\right)}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

10.2.3 Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

10.2.4 The analyst will not force the line through the origin, but have the intercept calculated from the five data points. The use of linear regression will not be used as a rationale for reporting results below the calibration range.

10.2.5 A linear least squares regression attempts to construct a linear equation of the form: $y= mx + b$, by minimizing the differences between the observed results(y_i, the response calculated from the constructed equation). The regression equation is:

$$y_i' = ax_i + b$$

where:

a = Regression coefficient or the slope of the line
b = The y-intercept
y_i' = Predicted (or calculated) response for the ith calibration standard.
x_i = Mass of analyte in the ith calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n} (y_i - y_i')^2$$

where n is the total number of calibration points. The regression calculations attempt to minimize the sum of squares, hence the name "least squares regression."

Weighting the sum of the squares of the differences can significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i \ (y_i - y_i')^2$$

where:

w_i = Weighting factor for the calibration standard (w=1 for unweighted least squares regression.
y_i = Observed instrument response (area or height) for the ith calibration standard.
y_i' = Predicted (or calculated) response for the ith calibration standard.
n = Total number of calibration standards.

10.2.6 Least Squares Equation (LSQ) weighting method to be used for calculation of least squares regression fits, either 1/x or $1/x^2$, gives increased importance to smaller concentrations and areas. LSQ weight can be applied to linear, quadratic, and cubic fits only.

10.2.7 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously

differentiable, and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it will be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.2.8 The statistical considerations in developing a non-linear calibration model require more data that the more traditional linear approaches described above. Linear regression employ five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards.

10.2.9 The "goodness of fit" of the polynomial equation is evaluated by calculating the weighed coefficient of the determination (COD);

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

.

y_{obs} = Observed response (area) for each concentration from each calibration standard.

$\bar{y}$ = Mean observed response from the initial calibration

Y_I = Calculated response at each concentration from the initial calibrations.

n = Total number of calibration points (6 points for quadratic equation)

p = Number of adjustable parameters in the polynomial.

10.2.10 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0 In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99.

10.2.11 Evaluation Standard Calculation: Evaluation standard must be $\leq$ 15%.

$$Eval = \frac{(DDD_A + DDE_A)}{(DDD_A + DDE_A + DDT_A)} * 100$$

A= Area Counts

11.0 PROCEDURE

11.1 Water Extraction (Method SW-846, 3510)

11.1.1 Pre-rinse all glassware to be used in the extraction with Methylene chloride (Pesticide Grade).

11.1.2 Mark the meniscus on the bottle for later determination of sample volume. (Refer to section 11.1.10) From the glass sample collection bottle, quantitatively transfer sample into a 2 liter separatory funnel.

11.1.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding one liter of Milli-Q water to a 2 liter separatory funnel.

11.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and from the glass sample collection bottle, quantitatively transfer half of the sample into a 2 liter separatory funnel and label MS, and transfer the other half of the sample into another 2 liter separatory funnel and label MSD. If there is no sample to perform a matrix spike/matrix spike duplicated, a laboratory control spike duplicate must be performed.

11.1.5 Check and adjust the extraction pH to between 5 and 9 with 10N sodium hydroxide and/or 1:1 sulfuric acid solutions.

11.1.6 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 0.5 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 0.5 mL of the pesticide spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike. If there is no sample to perform a matrix spike/matrix spike duplicate, notify project management. The default sample QC is to perform a laboratory control spike duplicate. A separate laboratory control spike for Toxaphene and tech-Chlordane will be extracted with each pesticide analysis (provided that these multi-component compounds are requested). Add 0.5mL of spiking solution of Toxaphene and tech-Chlordane and 0.5 mL of surrogate solution to the TOX/CHLOR laboratory control sample.

11.1.7 Add 60 mLs of methylene chloride to the sample's separatory funnel. The sample is extracted by the automated shaker. The shakers will shake the samples vigorously for two minutes. Continuous venting is provided by the special drilled cap located on the separatory funnel arm.

11.1.8 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Decant the lower layer into a 250 ml beaker. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ

mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and will include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.1.9 Repeat the extraction two more times using a fresh 60 mL portions of Methylene chloride.

11.1.10 Determine the original sample volume by filling the sample bottle to the mark (11.1.2) with water and transferring it to a "Class A" 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet.

11.1.11 Samples can potentially require cleanup prior to concentration/analysis. Refer to attachments; I, II, and III for sample cleanup procedure.

11.1.12 Refer to section 11.3 for sample concentration.

11.2 Soil Extraction (Method SW-846, 3545)

11.2.1 Preparing the extraction cell for use: Pre-rinse extraction cell (tube and caps) with Methylene chloride. Attach the screw fit tube cap of the soil extractor to the end of the tube without the serial number engraved on the side. Using a filter rod, push 1 Dionex ASE filter through the open end of the tube until they reside flush on the bottom of the screwed end.

11.2.2 Decant and discard any water layer from sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample more amenable to grinding.

11.2.4 Gummy, fibrous, or oily materials not amenable to grinding, shall be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample easier to mix. Wipe samples can be placed directly into the cell.

11.2.5 Weigh approximately 10 g of sample to the nearest 0.1 g in a 250-mL beaker and record the final weight. Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples shall be a free flowing powder. If sample is not free flowing, add more diatomaceous earth until the sample has a dry texture. This powder is mixed so that it will allow the sample to pass through a 1 mm sieve.

11.2.6 Transfer the ground sample to an extraction cell of appropriate size for the aliquot.

11.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10g of sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

11.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh two individual 10 grams aliquots of sample. Add drying agent. Transfer sample to extraction cell. If there is no sample available to perform a matrix spike/matrix spike duplicated, a laboratory control spike duplicate must be performed.

11.2.9 Fill the void in each of the extraction cells with clean sand. (Dionex ASE 200 Operator's Manual section 3.1.4 p.6).

11.2.10 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples, add 0.5 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 0.5 mL pesticide spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate). A separate laboratory control spike for Toxaphene and tech-Chlordane will be extracted with each pesticide analysis (provided that these multi-component compounds are requested). Add 0.5 mL of spiking solution of Toxaphene and tech-Chlordane and 0.5 mL of surrogate solution to the TOX/CHLOR laboratory control sample.

11.2.11 Attach the other cap to the extractor cell, making sure the tube is sitting flush on a hard surface so that no particulates get caught in the threads of the tube cap.

11.2.12 Place the extractor tube on the Dionex ASE with the serial number engraved end up on the top wheel. Place an appropriately labeled empty 60 mL VOA collection vial on the matching position on the bottom wheel. Schedule the Dionex ASE 200 and begin the cycle.

11.2.13 The Dionex ASE 200 extraction cycle:

Oven temperature: 100 º C Pressure: 1500 psi
Prepurge time: 0 minutes
Static time: 5 minutes
Heat: 5 minutes
Flush volume: 60%
Nitrogen purge: 60 sec. At 150 psi
Solvent A: 100%
Method rinse: ON
Static Cycles: 1
Extraction Fluid: (7:3) Methylene chloride : acetone

11.2.14 Samples can potentially require cleanup prior to concentration/analysis. Refer to attachments; I, II, and III for sample cleanup procedure.

11.2.15 Refer to section 11.3 for sample concentration.

11.3 Sample Concentration

11.3.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds full with Na_2SO_4. Rinse filter paper Na_2SO_4, funnel, K-D apparatus, and concentrator tube with Methylene chloride. Discard solvent rinse.

11.3.2 Quantitatively pour the extract through the filter and funnel seated on a 250mL Kuderna-Danish (K-D) apparatus complete with concentrator tube. Rinse the beaker or VOA vial three times with Methylene chloride. Add these rinses through the filter and funnel into the K-D. Add a boiling chip to the K-D flask prior to placing it on the heated water bath. Wet a three ball Snyder column with approximately 2 mL of Methylene chloride. Attach the Snyder column.

11.3.3 Place the K-D in the heated water bath so the concentrator tube is immersed in the water and the lower rounded surface of the K-D is bathed in steam. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood (set the knob of the temperature control to 4~5). It is critical that the analyst watch the extract as it distills. THE EXTRACT WILL NOT GO TO DRYNESS.

11.3.4 When the extract volume reaches approximately 5-7 mL, remove the K-D from the bath. Slightly tilt the apparatus and rotate to aid in solvent drainage from the Snyder column. Allow it to cool completely.

11.3.5 Remove the Snyder column and add 40 mL of hexane, then replace the Synder column. Adjust the temperature control of the water bath heater to 6~7. Mix the contents of the KD by slightly tilting and placing it upright several times using care so that the content does not spill out the Snyder column. Concentrate the extract to approximately 4 mL. Remove the K-D apparatus from the bath. As above, slightly tilt the apparatus and rotate to aid in solvent drainage. Allow the apparatus to cool completely.

11.3.6 Remove the Snyder column, rinse the ground glass joints with a small amount of hexane and then remove K-D flask. Add hexane to the concentrator tube so that the final volume is 10.0 mL. With a disposable Pasteur pipet draw up the extract and rinse the sides of the concentrator tube. Repeat this rinsing several times. (Note: If the sample extract appears highly colored or has a strong petroleum type odor, sample clean-up will be required.) Transfer about 1 mL of the extract to a labeled screw-cap injection vial, and the remainder into an amber storage vial. Record the final extract on the injection extraction bench sheet.

11.3.7 The sample extract is now ready for analysis. If samples are not analyzed immediately, store the sample extract in a refrigerator.

11.3.8 Samples can potentially require cleanup prior to analysis. Refer to attachments; I, II, and III for sample cleanup options.

12.0 SAMPLE ANALYSIS

12.1 Sample Sequence

12.1.1 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption will be a problem when the GC has not been used for a day or more. Therefore, the GC will be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Always run an acceptable instrument blank (hexane) prior to running any standards or samples.

12.1.2 Before initial calibration or sample analysis, an Evaluation standard must be performed. This standard will determine the break-down potential for the instrument. The standard will verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port. The compounds of interest are; Endrin, Endrin Aldehyde, Endrin Ketone, 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE. This standard must be analyzed at the beginning of the each analytical run and every twelve hours. The EVAL must pass QC requirements before analysis of standards or

samples. The break-down of the parent compounds; Endrin and 4,4'-DDT must be less than ≤15%.

12.1.3 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing Organochlorine Pesticides, prior to conducting any sample analysis. A CCV must be injected at the beginning of the sequence at intervals of no less than one per 20 samples injections or twelve hours (after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of each sequence. The variance of any given pesticide shall not be more than +/- 15% difference unless specified by project or program. A Toxaphene and tech-Chlordane CCV will be analyzed at the beginning or at the end of the sequence. QSM samples must have CCVs injected every ten samples for pesticides, Toxaphene, and tech-Chlordane.

12.1.4 Before the initial sample batch analysis, inject the sample preparation method blank to demonstrate that the instrument, as well as the extraction procedure, is free from contamination. All compounds must be below the method detection limit.

12.1.5 Samples can be directly injected after the successful analysis of the calibration curve, CCV, and method blank. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries must be within laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate, and laboratory control spike injections. If the sample shows interferences after analysis; such as sulfur or hydrocarbon contamination, cleanup will be required. Refer to attachment I, II, and III for sample cleanup options.

12.1.6 All positive detects in the associated sample must be confirmed by a second column. Positive detects are identified and quantitated using both columns. The results between primary and secondary columns must be within +/- 40 % difference, (if greater than 40% the target analyte is qualified with a "P"). If there is no evidence of chromatographic problems, then it is appropriate to report the lower result (method 8000C). Specific projects or programs will require that the higher of the two results be reported.

12.1.6.1 QSM: Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result. Qualify the data with a "Q" flag if target analyte is not confirmed by second column confirmation.

12.1.7 Each sample analysis must be bracketed with passing continuing calibration verification standards. If the continuing calibration verification standards fail to meet QC criteria, all samples that were injected after the last standard which met QC criteria must be re-analyzed.

12.1.8 The data acquisition software generates concentrations in ug/ml which can then be applied to the final calculation to give sample concentrations. Samples with results exceeding the calibration range are diluted accordingly in hexane and reanalyzed.

12.2 Sample Calculations

12.2.1 The concentration of each analyte in the sample is determined by the computer software. It calculates the amount of compound from the peak area, using the calibration curve. Linear regression of standard responses (R) against their known concentration (C in ug/ml), derives the following equation:

$$C = mR + b$$

12.2.2 Using the slope (m) and the intercept (b) from this equation, the concentration of the sample (ug/ml) is calculated and appears on the report page following the chromatogram.

Water samples:

$C_S = [(C_C)(V_E)(D)]/V_S$

Soil samples:

$C_S = [(C_c)(V_E)(D)]/[(W)(S)]$

Wipe samples:

$C = [(C_c)(V_E)(D)]$

Where: C_S = Concentration of sample in ug/L for waters and mg/kg on a dry weight basis for soils

C_c = Concentration from the curve (ug/ml)

C = Concentration in total ug

V_E = Total volume of sample extract (after concentration) in ml

V_S= Volume of water sample in liters

D = Dilution factor if extract was diluted

W = Weight of wet soil sample in grams

S = Total percent solids, expressed as: percent total solids/100

.

13.0 DATA ASSESSEMENT & ACCEPTANCE CRITERIA FOR QC MEASURES

13.1 Data Assessment & Acceptance Criteria for QC Measures

13.1.1 If the initial analysis of a sample or a dilution of the sample has a concentration of a particular pesticide that exceeds the calibration range, the sample must be re-analyzed at a dilution. If the initial sample exhibits interference such as; sulfur, petroleum hydrocarbons, or other baseline interference, the sample must under go sample clean-up. Refer to Attachments I and II. The method blank and laboratory control sample must accompany the sample during the clean-up procedure.

13.1.2 The qualitative identification of each pesticide determined by this method is based on retention time. This method employs a dual column comparison of dissimilar phases. The retention time is updated with the midpoint of each new calibration and is continually updated with the continuing calibration verification standards (CCVs). A new method ID is provided for each new sequence with updated retention times or alteration to the operating initial calibration method. If an alteration occurs (other than daily retention time updates), project management and the QA officer will be notified. A description of the alteration will be addressed in the client notes or case narrative.

13.2 Reporting Quantitative Analysis

13.2.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times are compared and the sample retention times have been made, the sample data can be reported. Assessments of all spiked, calibration control samples, and standards will also be scrutinized before reporting the data.

13.2.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors, and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 4.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point, the data is turned over to another qualified analyst for final validation. The second analyst confirms the results, electronically marks them validated, and signs his or her portion of the checklist (the checklist is included in this SOP). Finally, the validated results are made

available to the client services personnel in order for the data to be given to the client or appropriate agencies.

13.2.3 An electronic copy of the data is then filed and archived. The package includes; the sequence run log, a copy of the bench sheet, the LIMS run log, LIMS prep sheet, verification of calibration data, each sample's chromatogram. All the data is e initialed and dated by the analyst. Each sequence file header is labeled with the date of sequence.

13.3 Corrective Measures for Out-of-Control Data

13.3.1 When data is out of control, corrective actions are required. Certain situations will cause data deficiencies. If the non-conformities involve failing QC within the analytical sequence batch, the reanalysis of samples will eliminate any out of control data. If the out of control data is the result of instrument, malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data shall correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument will need cleaning and decontamination. The sample will need diluting or undergo sample clean-up procedures to reduce matrix affect. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions shall be taken for different QC-type failures or non-conformities (Table 3.).

13.3.2 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data can be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.

13.3.3 To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis so if a problem arises, re-analysis or re-extraction is an option.

13.3.4 If a sample is extracted passed holding time, the analyst must notify client services immediately. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur. If the client still requests sample analysis, the sample data is qualified for holding time violations.

13.3.5 If the non-conformities involve failing QC within the analytical sequence batch, the analyst must use professional judgment for the reanalysis of the extracts. The reanalysis will ensure that the instrument was functioning properly. If the results are passing, the non-conformities were associated with the instrument. If data is still outside QC criteria, re-extraction of the all samples associated with the QC samples must be performed (provide that there is sample available). If no sample is available for re-extraction, the samples are qualified accordingly.

13.3.6 If the out of control data is the result of instrument malfunctions, the analyst must evaluate and repair the instrument prior to reanalysis of the affected data. After the instrument maintenance and repair the corrective measures must be demonstrated by analyzing a Continuing Calibration Verification (CCV) before continuing sample analysis. Any maintenance and repair must be noted in the instrument logbook.

13.3.7 If sample matrix affect or contamination is the reason for poor data. The analyst will use professional judgment for the reanalysis of the extract. The extract can be diluted and reanalyzed. The sample can be re-extracted confirming matrix interference. The analyst can report and qualify the sample data. The analyst must supply explanation for the qualified data in the narrative or client notes.

13.3.8 If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors. Senior analysts have the authority to validate their own data.

13.3.9 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

13.3.9.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

13.3.9.2 In all instances, if results are reported from data that is out of control or unacceptable, that data is qualified accordingly. Once the client has been notified and he or she instructs us to report the data, it is flagged to indicate what type of nonconformity has occurred.

13.3.9.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data.

13.3.9.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating the nonconformance and to ensure that the proper corrective actions are employed.

14.0 WASTE MANAGEMENT

14.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).20.1

15.0 REFERENCES

15.1 USEPA, SW-846, 3rd Ed. Chapters 2&4, Method 8000C. March 2003.

15.2 USEPA, SW-846, "Organochlorine Pesticides by Gas Chromatography", Method 8081A Revision 1. December 1996.

15.3 USEPA, SW-846, "Organic Extraction and Sample Preparation", Method 3500B Revision 2. December 1996.

15.4 USEPA, SW-846, "Separatory Funnel Liquid-Liquid Extraction", Method 3510C Revision 3. December 1996.

15.5 USEPA, SW-846, "Pressurized Fluid Extraction", Method 3545 Revision 0. December 1996.

15.6 Department of Defense, "Quality Systems Manual for Environmental Laboratories". Version 3. January 2006.

ATTACHMENT I
SULFUR CLEANUP
METHOD 3660

1.0 SCOPE AND APPLICATION

This method covers the procedures used to eliminate sulfur interference from Pesticide extracts. Elemental sulfur is indicated by the presence of white crystals in the sample extract, or, upon analysis, a broadband interference from the solvent front.

2.0 REFERENCES

USEPA, SW-846, 3rd Ed. Method 3660. December, 1996.

3.0 APPARATUS AND MATERIALS

3.1 Mechanical shaker or mixer

3.2 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

3.3 Disposable culture tubes – 25 mL with Teflon-lined screwcaps.

3.4 Tetrabutylammonium (TBA) – sulfite reagent.

3.5 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212).

4.0 REAGENTS

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Organic-free reagent water.

4.3 Acetone, pesticide grade.

4.4 Hexane, pesticide grade.

4.5 2-Propanol, pesticide grade.

4.6 Tetrabutylammonium (TBA) sulfite reagent. (Aldrich Part # 86847-1EA-F).

4.7 Tetrabutylammonium hydrogensulfate (Aldrich Part # 155837-25G).

4.8 Sodium sulfite, (Aldrich Part# 239321-500G).

4.9 Prepare reagent by dissolving 3.39g tetrabutylammonium hydrogensulfate in 100ml organic-free water. To remove impurities, extract this solution three times with 20ml portions of hexane. Discard the hexane extracts. Add 25g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a PTFE-lined screw cap. This solution can be stored at room temperature for at least one month.

4.10 Nitric Acid, HNO_3, dilute

4.11 Copper powder, (UCT Part# ECCU01K)

5.0 PROCEDURE

5.1 TBA Solution

5.1.1 Pipet 1.0 mL of pesticide extract into a 25 mL culture tube.

5.1.2 Add 1.0 mL TBA – sulfite reagent and 2 mL iso-propanol to the tube and cap.

5.1.3 Shake for at least 1 minute. If the sample is colorless or if the initial color is unchanged, and if crystals are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100 mg portions until a solid residue remains after repeated shaking.

5.1.4 Add 5 mL distilled water and shake for at least 5 minutes. Allow the sample to stand for 5-10 minutes. Transfer the hexane layer to injection vials and/or storage vials.

5.1.5 Analyze the extracts by gas chromatography.

5.1.6 Verify that the TBA-sulfite is free from contamination by shaking out 1.0 mL of TBA – sulfite with 10 mL of hexane and analyze.

5.1.7 Process all quality control samples (e.g., spikes, blanks and duplicates) along with any samples.

5.1.8 Verify that recoveries of pesticides are greater that 80% by processing a standard (CCV) through the procedure.

5.2 Copper Powder

5.2.1 Remove oxides by treating the copper powder with dilute nitric acid, rinse with organic free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

5.2.2 Concentrate the sample to exactly 1.0ml or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

5.2.3 If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the concentration tube. Transfer 1.0ml of the extract to the calibrated centrifuge tube.

5.2.4 Add approximately 2g of cleaned copper powder to the centrifuge tube. Vigorously mix the extract and the copper powder for at least 1 minute on the mechanical shaker. Allow phases to separate.

5.2.5 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1 ml of extract. This step is necessary to prevent further degradation of the pesticides.

ATTACHMENT II
FLORISIL CARTRIDGE CLEANUP
METHOD 3620

1.0 SCOPE AND APPLICATION

1.1 Florisil, a registered trade name of U. S. Silica Co., is a magnesium silicate with basic properties. It is used to separate analytes from interfering compounds prior to sample analysis by a chromatographic method.

1.2 Florisil has been used for the cleanup of pesticide residues and other chlorinated hydrocarbons; the separation of nitrogen compounds from hydrocarbons; the separation of aromatic compounds from aliphatic-aromatic mixtures; and similar applications for use with fats, oils, and waxes. Additionally, Florisil is considered good for separations of steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates. Florisil cleanup can be accomplished using glass chromatographic column packed with Florisil or using solid-phase extraction cartridges containing Florisil. This method includes procedures for cleanup of sample extracts containing the following analyte groups:

Chlorinated hydrocarbons
Organochlorine pesticides
Organophosphates
Organophosphorus pesticides
PCBs
Phthalate esters
Nitrosamines
Nitroaromatics
Haloethers
Aniline and aniline derivatives

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for Florisil cleanup of solvent extracts of environmental samples using solid-phase extraction cartridges. The cartridge cleanup protocol uses solid-phase extraction cartridges containing 40 µm particles of Florisil (60 D pores). Each cartridge is rinsed with solvent immediately before use. The sample extract is loaded onto the cartridge which is then eluted with suitable solvent(s). A vacuum manifold is required to obtain reproducible results. The eluate will be further concentrated prior to gas chromatographic analysis.

3.0 INTERFERENCES

3.1 A reagent blank will be prepared and analyzed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples. The procedures for reagent purification outlined here will be considered to be the minimum requirements for use of this method. More extensive procedures shall be necessary to achieve lower levels of interferences for some analytes. During the evaluation of the cartridge clean-up procedure, phthalate esters were detected in the Florisil cartridge method blanks at concentrations up to 400 ng per cartridge. Therefore, complete removal of the phthalates esters from Florisil cartridges will not be possible.

4.0 APPARATUS AND MATERIALS

4.1 Vacuum manifold - VacElute Manifold SPS-24 (Analytichem International), Visiprep (Supelco, Inc.) or equivalent, consisting of glass vacuum basin, collection rack, funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500-mL sidearm flask fitted with a one-hole stopper and glass tubing. The manifold is required for use of the cartridge cleanup protocol.

5.0 REAGENTS

5.1 Eluting solvents - all solvents must be pesticide quality or equivalent.

5.2 Hexane, pesticide grade.

5.3 Methylene chloride, pesticide grade.

5.4 Acetone, pesticide grade.

5.5 Florisil cartridge pesticide check solution - Prepare a solution containing the following analytes in hexane:

BHCs
Heptachlor
BHC
Endosulfan I
Dieldrin
Endrin
4,4'-DDD
4,4'-DDT
Methoxychlor
Tetrachloro-m-xylene
Decachlorobiphenyl

6.0 PROCEDURE

6.1.1 Whenever Florisil is used to fractionate groups of target compounds (rather than to simply remove potential interference) it is critical that the specific fractionation scheme be validated using spiked solutions or spiked sample extracts that contain most or all of the analytes of interest. This is particularly important when the Florisil cartridge techniques are employed, as the differences between the various cartridge formats and manufacturers can affect the fractionation patterns. In addition, it will be useful to archive any fractions not originally intended for analysis in the event that the fractionation scheme chosen does not yield the intended results. Once the determinative analysis has been performed and demonstrates that the fractionation has been successful, such archived fractions can be disposed of in an appropriate manner. However, if the fractionation did not perform as intended, the analytes of interest contained in the archived fractions will be able to be analyzed or combined with the other fraction(s) for reanalysis.

6.1.2 Following Florisil cleanup, extracts will require further concentration and/or solvent exchange.

6.2 Cartridge Evaluation

6.2.1 The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

6.2.2 To check each new lot of Florisil cartridges before use, perform the following in duplicate.

6.2.3 Combine 0.5 ml of 2,4,5-Trichlorophenol solution in Section 5.5 and 0.5 ml of pesticide spiking solution in Section 5.6 in a 2ml autosampler vial.

6.2.4 Arrange the cartridges on the manifold in the closed-valve position.

6.2.5 Turn on the vacuum pump and set the vacuum to 10 in (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates can be controlled by opening and closing cartridge valves.

6.2.6 Condition the cartridges by adding 4 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

6.2.7 Slowly open cartridge valves to allow the hexane to pass through the cartridges.

6.2.8 Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed.

6.2.9 Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

6.2.10 Add the spiking solution in 6.2.3 to the Florisil cartridge.

6.2.11 Elute the cartridge with 9 ml of acetone/hexane (10/90, v/v). Collect the extraction in a 15 ml test tube.

6.2.12 Transfer extract to a concentrating thimble. Using the nitrogen blow down apparatus, concentration the extract down to 1ml and analyze by Method 8081A.

6.2.13 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110%, if the trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

6.3 Handling sample extracts

6.3.1 Most sample extracts will have to be concentrated to a smaller volume prior to the use of Florisil cleanup. The extract volume is a function of the analytical sensitivity necessary to meet the project objectives. The extract volume will also affect the ability of the Florisil to separate target analytes from potential interferences. Applying large extract volumes to the cartridges will cause poor results. Consult the appropriate extraction and determinative methods for the details on final extract volumes, extract concentration techniques, and solvent exchange procedures.

6.3.2 Reduce the sample extract volume to 10 mL prior to cleanup. The extract solvent will be hexane for these analytes. In most cases, given the sensitivity of the determinative methods, only 1 mL of the 10 mL extract needs to be subjected to the Florisil cleanup procedure. The remaining 9 mL will be archived for later use, if needed.

6.3.3 Place Florisil cartridges on manifold. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Condition the cartridges by adding 4 ml of hexane. Slowly open the cartridge valve and collect the eluate into the collection vial. Allow a few drops of hexane to pass through the cartridge to remove any air bubbles. Close valve and allow solvent to soak the entire sorbent bed for five minutes. Do not turn off vacuum.

6.3.4 Slowly, open the valve to allow the hexane to pass through the cartridge. Close valve when about 1 mm of hexane is remaining above the sorbent bed.

6.3.5 Add 1 ml of extract to the Florisil cartridge. Allow the extract to pass through the cartridge at a rate about 2 ml per minute.

6.3.6 Before extract goes below the surface of the sorbent bed, add 9 ml of hexane/acetone (90/10, v/v) to the cartridge.

6.3.7 After extract has been collected, concentrate sample down to 1 ml. Refer to Section 9.5 for concentration techniques.

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The following procedures are used to separate the organochlorine pesticides from PCBs.

6.3.8 Add 3 mL of hexane to the cartridge. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Allow the solvent to soak the sorbent bed for 1 minute or less. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 1 and it will contain the PCBs and a few of the organochlorine pesticides.

6.3.9 Close the cartridge valve, replace the collection vial, and add 5 mL of methylene chloride/hexane (26/74, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 2 and it will contain most of the pesticides.

6.3.10 Close the cartridge valve, replace collection vials, and add 5 mL of acetone/hexane (10/90, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 3 and it will contain the remaining pesticides.

6.3.11 As needed, perform a solvent exchange and adjust the final volume of the eluant to 1 ml.

7.0 QUALITY CONTROL

7.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A

recovery check must be performed using standards of the target analytes at known concentrations. The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

7.2 Organochlorine pesticides - To check each new lot of Florisil cartridges before use, perform the following in duplicate. Combine 0.5 mL of the 2,4,5-trichlorophenol solution, 1.0 mL of the pesticide solution, and 0.5 mL of hexane in a vial. Condition the cartridge and then perform the cartridge cleanup Elute the cartridge with 9 mL of acetone/hexane (10/90, v/v) only. Reduce the volume to 1.0 mL and analyze. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110 %, if the recovery of trichlorophenol is less than 5 %, and if no peaks interfering with the target analytes are detected.

ATTACHMENT II
GEL-PERMEATION CLEANUP
METHOD 3640A

1.0 SCOPE AND APPLICATION

1.1 Gel-Permeation Chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinylbenzene-styrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

1.2 General cleanup application – GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes; therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

1.3 Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue will ultimately reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem. GPC, operating on the principal of size exclusion, will not usually remove interference peaks that appear in the chromatogram since the molecular size of these compounds is relative similar to the target analytes. Separation cleanup techniques, based on other molecular characteristics (i.e. polarity), must be used to eliminate this type of interference.

1.4 This method is restricted for use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method is used to cleanup extracted samples with unwanted light and/or heavy compounds that interfere with final analysis of methods; 8081, 8082, 8270 and 8310.

2.2 Samples are extracted, then concentrated and prepared for GPC cleanup.

2.3 Determinations for which samples that are candidates for GPC cleanup are based on client specific, site specific, project specific requirements, specific characteristics of initial extract (e.g. color or odor) and/or interferences determined by initial extract analysis.

2.4 The column is calibrated and then loaded with the sample extract to be cleaned up. Elution is achieved with a suitable solvent(s) and collection times are adjusted based on the desired analysis. The collected fractions are then concentrated per guidelines for methods; 8081, 8082, 8270 and 8310.

3.0 INTERFERENCES

3.1 A reagent blank is to be analyzed for the compound of interest prior to the use of this method. The level of interferences must be below the estimated quantitation limits (EQL's) of the analytes of interest before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method will be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Gel-permeation chromatography system - Gilson GV-271 ASPEC, Gilson, INC or equivalent. All systems, whether automated or manual must meet the calibration requirements.

4.2 Chromatographic column -350mm x 21.20 mm 0 micron, Phenomenex p/n 00W-3035-PO or equivalent.

4.3 Guard column – (optional) 60mm x 21.20 mm 0 micron, Phenomenex p/n 03R-3035-PO or equivalent.

4.4 Ultraviolet detector –fixed wavelength (254 nm) with a semi-prep flow-through cell, Gilson 112 UV/VIS detector, Gilson, INC..

4.5 Strip chart recorder, recording integrator or laboratory data system, (Trilution and LIMS).

4.6 Syringe – 10ml with Luerlok fitting.

4.7 Syringe filter assembly, disposable – Puradisc 25TF sample filter assembly Whatman #6784-2510, 25mm and 1.0 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

4.8 Analytical balance – 0.01 g.

4.9 Volumetric flasks, Class A 5ml to 100ml

4.10 Graduated cylinders

4.11 Disposable glass culture tubes 13mm x 100mm, (Kimble 73500-13100).

4.12 Disposable glass collection tubes- (Kimax 51 25mm x 200mm, Kimble 45060-25200).

4.13 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

Reagents

4.14 Methylene chloride, Pesticide grade.

4.15 Cyclohexane, Pesticide grade.

4.16 N-Butyl chloride, Pesticide grade.

5.0 CALIBRATION

5.1 Preparation of standards is documented in the GPC standard logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards, once opened, expire within six months or sooner if routine QC indicates a problem, and are not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire within six months, and are not to exceed the opened date of the stock standard or the manufactures expiration date.

5.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Stock standards, once opened, expire within six months and are not to exceed the manufactures expiration date.

5.3 The following are commercially prepared certified stock standards

GPC Calibration Mix: Restek Part # 32019. (1ml ampul)
GPC Calibration Mix: Restek Part # 32023. (5ml ampul)

5.4 GPC Calibration Solution is a certified prepared standard in methylene chloride containing the following analytes (in elution order):

Compound	mg/mL
Corn oil	250
Bis (2-ethylhexyl) phthalate	10
Methoxychlor	2.0
Perylene	0.2
Sulfur	0.8

Table 1.0 GPC Calibration Standard

Standard	Component Name	Conc. (mg/ml)	STD Volume (ml)	Final Volume (ml)	Final Conc. (ug/ml)
GPC STD	Corn Oil	250	5.0	50.0	25000.0
	Bis(2-ethylhexyl)pht.	10	5.0	50.0	1000.0
	Methoxychlor	2.0	5.0	50.0	200.0
	Perylene	0.2	5.0	50.0	20.0
	Sulfur	0.8	5.0	50.0	80.0

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND EXTRACTION PROCEDURES

6.1 Follow guidelines listed in section 8.0 in SOP 8081 Rev. 6 for sample collection, preservation and handling.

7.0 PROCEDURE

7.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 72°F.

7.2 **GPC Setup**

Column Preparation:

7.2.1 Using manual control options in Trilution, set the system to equilibrate for at least 30 minutes prior to starting a run or priming.

7.2.2 If system has not been used on a regular basis, prime all lines and pumps either manually or using the automated system following guidelines listed in the Trilution help manual.

7.3 Calibration of the GPC Column

7.3.1 Place approximately 6 to 7 mls of the calibration solution in a disposable culture tube. Place tube in position 1 on the sample tray. Set Trilution to run a calibration method with an inject volume of 5.50ml.

7.3.2 Following are criteria for evaluation of the UV chromatogram for column condition.

7.3.3 Peaks must be observed and will be symmetrical for all compounds in the calibration solution.

7.3.4 Corn oil and phthalate peaks must exhibit >85% resolution.

7.3.5 Phthalate and methoxychlor peaks must exhibit >85% resolution.

7.3.6 Methoxychlor and perylene peaks must exhibit >85% resolution.

7.3.7 Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

7.3.8 Nitroaromatic compounds are particularly prone to adsorption. For example, 4-nitrophenol recoveries can be low due to a portion of the analyte being discarded after the end of each collection time. Columns are to be tested with the semivolatiles matrix spiking solution. GPC elution will continue until after perylene has eluted or long enough to recover at least 85% of the analytes, whichever time is longer.

7.3.9 Calibration for Semivolatiles – Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Collection can be stopped before sulfur elutes. Each laboratory is required to establish its specific time sequences.

7.3.10 Calibration for Organochlorine Pesticides/PCBs – Determining the elution times for the phthalate, methoxychlor, perylene and sulfur. Choose a dump time which removes >85% of the phthalate, but collects >95% of the methoxychlor. Stop collection after the elution of perylene, but before sulfur elutes.

7.3.11 Calibration for Polynuclear Aromatic Hydrocarbons – Determine the elution times for corn oil, phthalate and perylene. Start elution

collections just before the elution of bis (2-ethylhexyl) phthalate and end collection after the elution of perylene.

7.3.12 Verify the flow rate by letting the collection column eluate for 10 minutes in a graduated cylinder and measure the volume, which will between 45-55ml (4.5-5.5 ml/min). If flow rate is outside of this range, corrective action must be taken (as described above). Once flow rate is within the ranges of 4.5-5.5 ml/min, record the column pressure (6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If flow rate and/or column pressure do not fall within the above ranges, the column or pre-column are suspected. A UV trace that does not meet the criteria in section 7.2.2.2 would also indicate that the column or pre-column is responsible the poor performance.

7.3.13 Reinject the calibration solution after appropriate collect and dump cycles have been set and the solvent flow and column pressure have been established.

7.3.14 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed can be used to indicate problems with the system during sample processing.

7.3.15 The retention times for bis (2-ethylhexyl) phthalate and perylene must not vary more than ± 5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by:

7.3.15.1 Poor laboratory temperature control or system leaks.

7.3.15.2 An unstabilized column that requires pumping methylene chloride through it for several more hours.

7.3.15.3 Excessive laboratory temperatures, causing outgassing of the methylene chloride.

7.3.16 Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by whatever detectors will be used for the analysis of future samples. Exchange the solvent if necessary. If the blank exceeds the estimated quantitation limit of the analytes, pump additional methylene chloride through the system for 1 to 2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

7.3.17 A hard copy calibration report will follow the samples in the sample prep folder.

8.0 EXTRACT PREPARATION

8.1 Adjust the extract volume to 5 ml. The solvent extract must be primarily methylene chloride. All other solvents, e.g. 1:1 methylene chloride/acetone, must be concentrated to 1 ml (or as low as possible if a precipitate forms) and diluted to 5 ml with methylene chloride. Thoroughly mix the extract before proceeding.

8.2 Filter the extract through a 1 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 ml syringe. Draw the sample extract through the filter assembly and into the 10 ml syringe. Disconnect the filter assembly before transferring the sample extract into a small glass container, e.g. a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into a syringe without the filter assembly, attach the filter assembly and force the extract through the filter and into the glass container. The latter is the preferred technique for viscous extracts or extracts with a lot of solids. Particulate larger than 5 microns can scratch the valve, which can result in a system leak and cross-contamination of sample extracts in the sample loops.

NOTE**:** Viscosity of a sample extract can not exceed the viscosity of 1:1 water/glycerol. Dilute samples that exceed the viscosity.

9.0 SCREENING THE EXTRACT

9.1 Screen the extract to determine the weight of dissolved residue by evaporating a 100 µL aliquot to dryness and weighing the residue. The weight of dissolved residue loaded on the GPC column cannot exceed 0.500 g. Residues exceeding 0.500 g will very likely result in incomplete extract cleanup and contamination of the GPC switching valve (which results in cross-contamination of sample extracts).

9.1.1 Transfer 100µL of the filtered extract from section 8.2 to a tared aluminum weighing dish.

9.1.2 A suggested evaporation technique is to use a heat lamp. Set up a 250 watt heat lamp in a hood so that it is 8 ± 0.5cm from a surface covered with a clean sheet of aluminum foil. Surface temperature will be between 80-100°C (check temperature by placing a thermometer on the foil and under the lamp). Place the weighing dish under the lamp using tongs. Allow it to stay under the lamp for 1 minute. Transfer the weighing dish to an analytical balance

or a micro balance and weigh to the nearest 0.1mg. If the residue weight is less than 10mg/100µL, then further weightings are not necessary. If the residue weight is greater than 10mg/100µL, then determine if constant weight has been achieved by placing the weighing dish and residue back under the heat lamp for 2 or more additional 0.5 minute intervals. Reweigh after each interval. Constant weight is achieved when three weights agree within ±10%.

9.1.3 Repeat the above residue analysis on a blank and a spike. Add 100µL of the same methylene chloride used for the sample extraction to a weighing dish and determine residue as above. Add 100µL of a corn oil spike (5g/100ml) to another weighing dish and repeat the residue determination.

9.1.4 A residue weight of 10mg/100µL of extract represents 500mg in 5ml of extract. Any sample extracts that exceed the 10mg/100µL residue weight must be diluted so that the 5ml loaded on the GPC column does not exceed 0.500g. The following is a calculation that will be used to determine what dilution is necessary if the residue exceeds 10mg.

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{\text{10mg maximum}}{\text{X mg of residue}}$$

Example

$$\text{Y ml taken for dilution} = 5 \text{ ml final volume} \times \frac{\text{10mg maximum}}{\text{15 mg of residue}}$$

Y ml taken for dilution = 3.3 ml

Therefore, taking 3.3 ml of sample extract from step 7.4.2 and diluting it to 5ml with methylene chloride will result in 5 ml of diluted extract loaded on the GPC column that contains 0.500 g of residue.

NOTE**:** This dilution factor must be included in the final calculation of analyte concentrations.

10.0 GPC CLEANUP

10.1 Calibrate the GPC at least once per week following the procedure outlined in section 7.3. Ensure that UV trace requirements, flow rate and column pressure criteria are acceptable. Also, retention time shift must be <5% when compared to retention times in the last calibration UV trace.

10.2 If these criteria are not met, follow appropriate maintenance methods to try to regain resolution.

10.3 Load sample vials with 5 ml of filtered extract from section 7.4.2. The Method Blank (MB) and Laboratory Control Standard (LCS) must be ran through the GPC process from each prep batch that the samples originated from.

NOTE**:** The number of samples from each prep batch that are put through GPC will vary by client, location and project specifications

10.4 Set the GPC up to run cleanup of the samples, verifying that collection and dump times correlate with the calibration times for each analysis specified in section 7.3. Multiple methods can be used on each GPC run. Follow software specific guidelines for setting up a GPC run.

NOTE**:** It will be necessary to run multiple rinse methods between samples to ensure no cross-contamination between particularly dirty samples

10.5 Monitor sample volumes collected. Changes in sample volumes collected will indicate one or more of the following problems:

10.5.1 Change in solvent flow rate caused by channeling in column or changes in column pressure.

10.5.2 Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

10.5.3 Leaks in the system or significant variances in room temperature.

10.6 Concentrate the extract by following the methods listed in the sections for Sample Concentration for methods; 8081, 8082, 8270 and 8310.

11.0 QUALITY CONTROL

11.1 The analyst will demonstrate that the compound(s) of interest are being quantitatively recovered before applying this method to actual samples.

11.2 For sample extracts that are cleaned up using this method, the associated quality control samples must also be processed through this clean up method.

11.3 Some brands of methylene chloride can contain high levels of acid (HCl). Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer.

11.4 If the pH of the water layer is ≤5, filter the entire supply of solvent through a 2 in x 15 in glass column containing activated basic alumina. This column will be sufficient for processing approximately 20-30 liters of solvent. Alternatively, find a different supply of methylene chloride.

Table 3
Summary of Method Quality Objectives for Method 8081- Pesticides

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Initial Calibration	Initial Calibration prior to sample analysis. 1. RSD for each analyte ≤ 20% 2. Linear – least squares regression r ≥ 0.995. 3. Non-linear regression $r^2 \geq 0.99$. (6 points shall be used for second order)	$r^2 \geq 0.990$, RSD ≤20%, r ≥0.995, mean RSD ≤ 20%	Linearity must fit one of acceptance criteria, or the curve must be reanalyzed.
Initial Calibration Verification (ICV)	After initial calibration	Difference ≤15% QSM: 80-120%	If ICV falls outside QC criteria, reanalysis must take place. If ICV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". QSM: No samples will be analyzed until the problem has been corrected.
Continuing Calibration Verification (CCV)	Every twelve hours or every twenty samples (which one comes first) QSM: Every ten samples	Difference ≤15% QSM: ≤20%	If CCV falls outside QC criteria, reanalysis must take place. If CCV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with "Z". Exception to the above, if the acceptance limits are exceeded high and the analyte is not detected in the sample the verification standard has passed (analyte would have been detected if present). QSM: No samples will be analyzed until the problem has been corrected.
Retention Time Window (RTW)	Retention Times will be set using the midpoint standard in the ICAL or the RT in the CCV run at the beginning of each analytical shift.	Shift less than ± 3 times the absolute Standard Deviation from the 72 hour RTW study, with a minimum width of 0.03 minutes	QSM: No samples shall be run without a verified retention time window at the initial verification. For CCVs, apply a "Q" to all results for analytes outside the retention time window.
MDL Study/MDL Verification Check	At initial set-up and annually for MDL Study. MDL Verification Checks shall be performed quarterly.	MDL checks must produce a signal at least 3 times the instrument's noise level.	Samples can not be analyzed without a valid MDL. QSM: Run MDL verification check at higher (2x MDL).

Table 3
Summary of Method Quality Objectives for Method 8081- Pesticides

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
MRL Level Verification Check standard at Reporting Limit.	Beginning and End of 12 hr. sequence or program specified.	70-130% or project specific/client limits	For results > 130% apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at the end and acceptable do not reject data.
Evaluation Standard	Beginning of a analytical sequence and every twelve hours	Breakdown for Endrin and 4,4'-DDT must be ≤15%	Correct problem and repeat.
Method Blank (MB)	1 per sample batch ≤ 20 samples of the same matrix	Analytes must not he higher than the highest of the following: 1/2 MRL, or 5% of the regulatory limit, or 5% of the associated sample concentration. QSM = ½ MRL	If sample is available and within holding times, sample associated with method blank needs to be reprepped. If no sample is available, qualify the data with a "B" to all associated positives when less than 5X blank concentration. QSM: Apply "B" to all results for the specific analytes in all samples in the associated preparatory batch.
Laboratory Control Sample (LCS)	1 per sample batch ≤ 20 samples of the same matrix	In-house limits, or program or client specified limits	If LCS fails percent recoveries. Re-analyze the LCS. If LCS recoveries are still outside QC control limits, if there is sample remaining, the sample batch must be reprepped. Qualify the failing analytes with a "Q". QSM: Correct problem, then repep and reanalyze the LCS and all samples in the associated batch for the failed analytes in the samples, if sufficient sample is available.
Matrix Spike (MS)	1 per sample batch ≤ 20 samples of the same matrix	In-house Limits, or program or client specified limits	No action is taken based on MS recovery alone, use of professional judgement. For recoveries outside QC criteria, qualify out lying compound with "M". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.
Matrix Spike Duplicate (MSD)	1 per sample batch ≤ 20 samples of the same matrix	In-house Limits, or program or client specified limits	No action is taken based on MSD results alone, use of professional judgement. If RPD is outside QC criteria, then qualify the out lying compounds with "Y". QSM: For the specific analyte(s) in the parent sample, apply "J" if acceptance criteria are not met.

Table 3
Summary of Method Quality Objectives for Method 8081- Pesticides

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Surrogates	Every sample and QC	In-house Limits, or program or client specified limits	Rerun sample. If no apparent matrix interference noticed re-extract sample. If no sample is available, qualify the surrogate with "S". QSM: For QC and field samples; correct problem, reprep and re-analyze all failed samples or failed surrogates in the associated batch, if sufficient sample material is available. If no sample available, field samples apply "J" to the specific sample, QC samples apply "Q" to all associated samples
Target Analyte Confirmation	Whenever a positive is detected, check agreement between primary and secondary columns.	RPD $\leq$ 40% QSM: Discuss in case narrative about qualified data.	**If not present**; QSM: Report positive value with a "Q" qualifier. Non-QSM: Do not report value. **If present and RPD $\leq$40%** QSM: Report higher of the two values. Non-QSM: Reporting the lower of the two values may be appropriate. **If present and RPD >40%** QSM: Report higher value with a "J" qualifier. Non-QSM: Report lower of the two values, may be appropriate, with a "P" qualifier.

Table 4

Analytical Run# **8081 Pesticide Analysis Data Review Checklist**

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the Pesticide initial calibration performed using a minimum of five varying standard concentration levels on two dissimilar columns?	Lowest standard at or near MRL					
b. Was there initial calibration performed using a minimum of five varying standard concentration levels for Toxaphene and tech-Chlrodane.						
c. Is the variation between calibration response factors for all concentration levels <20% RSD, is r >.995, or r^2 > 0.990 for the regression line?	RSD <20%, r >.995, r^2>0.990					
d. Was each ICAL uniquely identified (i.e. Standard Number)?						
e. Was an initial calibration blank (ICB) analyzed?						
f. Was there an Evaluation standard injected prior to initial calibration?	Breakdown ≤15%					
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the recoveries for the ICVs within program limits?	%Recovery					
c. Was the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run?						
b. Were there a CCV for Toxaphene and tech-Chrodane at the beginning or at the end of . the analytical sequence?						
c. Were the recoveries for the CCVs within program limits?	%Recovery					
d. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8081 Pesticide Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?	In-house limits or client specified limits					
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Was there a Toxaphene and tech-Chlordane LCS preformed						
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a . frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	In-house limits or client specified limits					

Table 4
8081 Pesticide Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. florisil, GPC, and sulfur treatment)	Florisil, GPC, and/or Sulfur Treatments					
f. Was there a hexane injection preformed prior to sample analysis?						
g. Was there a priming standard injected prior to sample analysis?						
h. Was there an Eval standard injected prior to sample analysis and every twelve hours?	Breakdown $\leq$15%					
9. RECORDS AND REPORTING						
a. Is the Analytical Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, analytical data, and method transfered to PDF format?						
b. Are all chromatograms stamped with initials and date?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier in LIMS and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

CT Laboratories

Title: **Soil and Waste pH - Electrometric Measurement**

SOP Number: CC-24B

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance *Date*

_____
Laboratory Director *Date*

SOP Manual Control Number:_____

1.0 **SCOPE AND APPLICATION**

1.1 This is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

2.0 **METHOD SUMMARY**

2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured electrometrically using a combination electrode. The measuring device is calibrated using a series of standard solutions of known pH.

3.0 DEFINITIONS

3.1 Duplicate Analysis: Two aliquots of a given sample are analyzed. The difference is then determined from the two results and compared to the lab control limit.

4.0 HEALTH AND SAFETY

4.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTI Health and Safety Manual).

5.0 **CAUTIONS**

5.1 Make sure that the probe is rinsed well with DI water between readings.

6.0 **INTERFERENCES**

6.1 Samples with very low or very high pH may be difficult to read accurately. For samples with a pH of >10, the measured pH may be incorrectly low. This error is due to the high sodium content of some wastes. If the solid state electrode is used, and if kept clean and maintained regularly, this interference will not be significant. A low sodium error electrode should be used for these particular samples.

6.2 Coatings of oily material or particulate matter can impair electrode response. Insert electrode into beaker of 100% methanol and stir for 30 seconds. Thoroughly rinse probe with DI water to remove the coatings.

6.3 Temperature may affect the electronic measurement of pH in two ways. Electrode output will vary with changes in temperature. This problem is controlled by using the solid state probe which is temperature compensated. The slope change will be compensated for automatically. The second source of temperature problems is the change of pH due to changes in the sample as the temperature changes. This error is sample dependent and cannot be controlled. The temperature of the sample should therefore be measured and recorded at the time of pH measurement.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel need to demonstrate capability by completing a valid IDC, initial demonstration of capability, study prior to performing analyses on actual samples

8.0 APPARATUS AND MATERIALS

8.1 pH meter, Orion 920A microprocessor based Solid State pH Meter
8.2 Electrode: Orion Triode with BNC connector or equivalent
8.3 Beaker, 100-150 ml borosilicate glass.
8.4 Stir plate with stir bar (teflon coated).
8.5 Denver Analytical Balance: capable of measuring 0.1 gm.
8.6 25 ml Graduated Cylinder
8.7 Reagents
- 8.7.1 pH 4.00 Buffer solution. Store in wet chem lab under sink
- 8.7.2 pH 7.00 Buffer solution. Store in wet chem lab under sink
- 8.7.3 pH 10.00 Buffer solution. Store in wet chem lab under sink
- **8.7.4** Internal Fill solution. Store on shelf in wet chem lab.
- **8.7.5** 7.00 buffer/potassium chloride solution: into a 1 L volumetric flask, dissolve 4 g of potassium chloride (KCl) and bring up to volume with pH 7.00 buffer. Store on shelf in wet chem lab.

9.0 INSTRUMENT OR METHOD CALIBRATION

9.1 Calibrate meter daily.
9.2 Check internal reference solution and fill if necessary.
9.3 Press Calibrate. The word CALIBRATE is displayed.
9.4 After a few seconds ENTER NO. BUFFERS is displayed. Press 3 and then press Yes.
9.5 When the BUFFER 1 prompt appears, place the electrode into the pH 4.0 buffer. Always use fresh buffers daily.
9.6 When the electrode signal has stabilized, the prompt READY CAL AS will appear. Record the pH displayed in the pH calibration log book. Press Yes. Rinse the probe.
9.7 Repeat steps 3 and 4 for the pH 7.0 and 10.0 buffers.
9.8 The slope of the line should appear. This appears *very briefly* after you press YES following the pH 10 buffer. Record the slope in the pH calibration log book.
9.9 The meter will automatically go to the MEASURE mode.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 Sample should be analyzed as soon as possible, preferably at the time of collection. Many clients have us measure the pH even though they would be considered past hold time by the time they get to the lab. On the sample reports, lab pH designates these samples as lab pH.
10.2 For liquid samples, collect a minimum of 125 mls of sample in a plastic or glass container.

10.3 High purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere. To minimize this effect, fill sample containers completely and keep sealed prior to analysis.

11.0 SAMPLE PREPARATION AND ANALYSIS

Solid Samples

11.1 For solid samples, add an equal amount of DI water and sample to a clean beaker. The routine volume is 20mL of DI water and 20g of sample. If the consistancy is still too thick, add more DI water and note that on the log sheet. Place a stir bar into the beaker, place on a stir plate and allow sample to stir for 5 min.

11.2 Alternately, if processing a large number of samples, omit the stir bar, cap the sample containers, place the samples on a bench-top shaker and agitate for 5 minutes.

11.3 Let the soil suspension stand for about 1 hour to allow most of the suspended clay and other fine material to settle out.

11.4 Place the electrode into the sample, immersing to a sufficient depth to cover the sensing elements and give good electrical contact.

11.5 After stabilization, record the pH of the sample from the meter. Report pH to the nearest 0.1 pH units. For every pH measurement, record the sample temperature to the nearest degree C on the benchsheet.

11.6 Rinse the probe after every sample.

11.7 Store probe in the vial of 7.00 buffer/potassium chloride solution with meter on standby. DO NOT turn the meter off. DO NOT unplug the probes or the meter from their respective sockets.

Waste Samples

11.8 For waste samples, add an equal amount of DI water and sample to a clean beaker. The routine volume is 20mL of DI water and 20g of sample. Additional dilutions are allowed if working with hydroscopic wastes, salts or other problematic material, noting that on the log sheet. Place a stir bar into the beaker, place on a stir plate and allow sample to stir for 5 min.

11.9 Let the waste suspension stand for about 15 minutes to allow most of the suspended material to settle out.
Note: If the waste is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it comes in contact with an oily material.

11.10 Continue as with Steps 11.4 through 11.7 above.

12.0 TROUBLESHOOTING AND MAINTENANCE

12.1 Refer to Benchtop pH/ISE meters Model 920A Instruction Manual for further instructions.

13.0 **DATA ACQUISITION, CALCULATION AND REDUCTION**

13.1 There are no additional data calculations required for pH.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 A computer with StarLIMS

15.0 **DATA MANAGEMENT AND RECORD MANAGEMENT**

15.1 After data has been entered into LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.

15.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

15.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.

15.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

16.0 **REFERENCES**

16.1 *Methods for Chemical Analysis of Water and Wastes,* U.S. EPA, 1983, EPA-600/4-79-020, Method 150.1.

16.2 *Test Method for Evaluating Solid Waste,* U.S. EPA SW 846 3rd Edition, Method 9045.

16.3 Benchtop pH/ISE meters Model 920A Instruction Manual, Orion.

17.0 **QUALITY CONTROL/ QUALITY ASSURANCE**

17.1 Duplicate analysis is done every 20 samples per matrix. A sample is mixed well and divided into two equal aliquots with each aliquot being measured, the range between the two aliquots is calculated, and must be less than or equal to 0.1 pH.

17.2 When the probe is being calibrated, record the pH value from the meter in the calibration logbook. In addition, at the end of calibration record the slope value and the temperature of the buffers. The acceptable range for the electrode slope is 90-110%. Generally, the slope value is closer to 95-105%. Slope values below 92% indicate that the probe may need cleaning. Follow the manufacturer recommendations, then recalibrate.

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Sample duplicate (DUP)	One (1) per 20 samples per matrix	Must be < 0.1 pH difference.	Either re-calibrate or re-homogenize sample.

Data Validation Checklist

LIMS #:	Method: pH EPA 150.1			
Analysis Date	Analyst / Data Interpretor	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Was the calibration slope recorded on the bench sheet?	---					
2. Were duplicates analyzed at the appropriate frequency?	1 every 20 samples of the same matrix					
3. Were the duplicates within acceptable limits?	Differ by $\leq$ 0.10 pH units					
4. Are all samples on the job lists accounted for?	---					

CT Laboratories
Baraboo Laboratory Divison

Title: **Acid Digestion of Waters for Dissolved or Total Recoverable Metals**

SOP Number: **6205B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare surface water and groundwater samples for analysis. Samples prepared by this method may be analyzed by AAS or ICP for the following metals:

Aluminum	Calcium	Magnesium	Silver
Antimony	Chromium	Manganese	Sodium
Arsenic	Cobalt	Molybdenum	Strontium
Barium	Copper	Nickel	Thallium
Beryllium	Iron	Potassium	Vanadium
Cadmium	Lead	Selenium	Zinc
Boron*			

*Analyze by ICP Only

2.0 METHOD SUMMARY

2.1 Total recoverable metals - The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substantially reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

2.2 Dissolved metals - The sample is filtered through a 0.45-µm filter at the time of collection and the liquid phase is then acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 Reagent Blank - A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.3 LCS (Laboratory Control Sample)- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the digestion.

3.4 PB or MB (Prep Blank or Method Blank)- A reagent blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation process. A minimum of one MB is prepared per batch and is analyzed at the beginning of an analytical batch.

3.5 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every batch. Failure to meet criteria may be due to poor

recovery during the preparation method or to matrix interference within the digestate. To be considered acceptable, MSD's must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD and may be used as acceptance criteria for duplicate analysis.

3.6 Detection Limit (DL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

4.0 HEALTH AND SAFETY

4.1 Gloves, protective eyewear, and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

5.1 Do not allow samples to evaporate to dryness. If the samples do go to dryness, they must be reprepped.

5.2 Use caution when adding acid to hot samples. Sample can foam and splash when the acid is being added.

6.0 INTERFERENCES

6.1 There are no interferences

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis should be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 All personnel performing this analysis should have a valid IDC prior to performing preparation on samples.

8.0 APPARATUS AND MATERIALS

8.1 Environmental Express Hotblock Digestion Apparatus: To maintain a temperature of 90-95C.

8.2 50 mL Graduated hotblock digestion tubes, with watch glasses and storage caps.

8.3 Qualitative filter paper and filter funnels.

8.4 Filtermate Teflon digestion tube filters: Environmental Express

8.5 Thermometer: For monitoring temperature of the block at beginning and end of digestion process.

8.6 REAGENTS

8.6.1 ASTM Type II Water (ASTM D1193): Water should be monitored for impurities.

8.6.2 Concentrated Nitric Acid, trace metals grade (HNO_3): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given

element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with another bottle from the same lot. If the entire lot is contaminated, then any remaining acid from the lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained.

8.6.3 Concentrated Hydrochloric Acid, trace metals grade (HCl): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with another bottle from the same lot. If the entire lot is contaminated, then any remaining acid from the lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained.

8.6.4 Inorganic Ventures Spike 1

8.6.5 Spike Standard- SPEX # SPIKE-1-500

8.6.6 Molybdenum standard- 1000 mg/L

8.6.7 Ultra Custom Standard- ICUS 192

8.6.8 Potassium standard- 10,000 mg/L

8.6.9 Boron standard- 1000 mg/L

9.0 INSTRUMENT OR METHOD CALIBRATION

9.1 There is no calibration requried

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

	Total Recoverable Metals	Dissolved Metals
Preservative	pH <2 HNO_3	filtered, pH <2 with HNO_3
Hold Time	180 days	180 days

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Total Recoverable Metals

11.1.1 Measure 50 mL of well-mixed sample into a clean, labeled hot-block digestion tube. For blanks use same amount of Type II water.

11.1.2 For samples to be spiked (MS, MSD, LFB/LCS), add the appropriate amount of spiking solution to sample (see Pre-digestion Spikes-LCS & LFB chart attached).

11.1.3 Add 1-mL conc. HNO3 and 2.5 mL conc. HCL. (For samples to be analyzed by GFAA omit the HCL).

11.1.4 Cover with a plastic ribbed watch glass for samples that may react strongly to acid or heat. Lower the tube rack into hot-block and heat at 90-95^0 C until the volume has been reduced to 5-10 mL. Do not allow sample to go dry.

11.1.5 Place thermometer in one block location, record temperature in the

sample prep book after 30 min, for the initial block temperature recording. Record again at the end of the digestion process.

11.1.6 Remove each tube from block using the tube racks provided and allow them to cool. Dilute to original volume and let sample sit to allow solids to settle out. If solids do not settle readily and remain suspended throughout sample, then it will be necessary to filter prior to analysis. Using the Environmental Express tube filters, push the filter down through the sample till it sits on the bottom. Alternately, filter through acid rinsed filter paper, rinsing at least 3 times into the funnel till filled to the 50 mL line. When using the tube filter fill tube to the 50 mL line first. If using filter and funnel, dilute to 50 mL line after.

11.1.7 Cap sample, taking care not to touch underside of cap or inside of container. To minimize contamination and for safety purposes appropriate gloves must be worn at any point where sample solution is being transferred. If gloves become stained and dirty, discard and replace, as the surface of the gloves themselves may also transfer contaminants.

11.1.8 If the digestate is clear and without solids distributed throughout the liquid portion, sample aliquots may then be poured off the top for analysis. If not, then filtration will be necessary prior to analysis. Follow instruction provided with filter-mate filters.

11.2 Procedure - <u>Dissolved Metals</u>

11.2.1 No digestion is necessary as long as the sample has been filtered prior to preservation, and the acid concentrations are adjusted to match those of the standards.

11.2.2 Adjustment of Samples for dissolved GFAA metals: None

11.2.3 Adjustment of Samples for dissolved FLAA/ICP metals:

11.2.3.1 Add 4.8 mL conc HNO_3 for every 100 mL of sample

11.2.3.2 Add 5.0 mL conc HCl for every 100mL of sample

12.0 TROUBLE SHOOTING AND MAINTENANCE

12.1 Refer to the Environmental Express manual for troubleshooting and maintenance.

13.0 DATA ACQUISITION, CALCULATIONS AND REDUCTION

13.1 There are no calculations associated with this procedure.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS.

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Data is recorded in the Metals digestion log.

15.2 Prep data is entered into LIMS, and then the batch sheet is given to the metals prep analyst.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 For each batch of samples processed, a method blank (Type II water and reagents) should be carried throughout the entire sample preparation procedure.

16.2 Prepare one MS/MSD per matrix per 20 or fewer samples per digestion batch.

16.3 Prepare one LCS (lab control sample) for each digestion batch by spiking reagent water with the same amount of analyte used for the MS/MSD. The LCS is prepared from one or more commercially prepared reference standards but must be from a source different from that used to prepare the calibration standards for a given instrument. The LCS solution is carried through the entire digestion procedure. Prepare one LCS for every 20 samples on a given matrix per day.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*. USEPA SW-846, 3rd Edition, 1992, Method 3005A.

Pre-Digestion Matrix Spike Prep for ICP/FLAA

Element	Spike Amt mL	Spike Stock	Stock Conc mg/L	Final Vol mL	Expected Conc. ug/L
Aluminum	1	**A**	200	50	4000
Antimony	1	**A**	50	50	1000
Arsenic	1	**A**	200	50	4000
Barium	1	**A**	200	50	4000
Beryllium	1	**A**	5	50	100
Boron	0.1	**D**	1000	50	2000
Cadmium	1	**A**	5	50	100
Calcium	0.5	**C**	20,000	50	200000
Chromium	1	**A**	20	50	400
Cobalt	1	**A**	50	50	1000
Copper	1	**A**	25	50	500
Iron	1	**A**	100	50	2000
Lead	1	**A**	50	50	1000
Manganese	1	**A**	50	50	1000
Magnesium	0.5	**C**	10,000	50	100000
Molybdenum	0.1	**B**	1000	50	2000
Nickel	1	**A**	50	50	1000
Potasium	0.5	**E**	10,000	50	100000
Selenium	1	**A**	200	50	4000
Silver	1	**A**	5	50	100
Sodium	0.5	**C**	10,000	50	100000
Thallium	1	**A**	200	50	4000
Vanadium	1	**A**	50	50	1000
Zinc	1	**A**	50	50	1000

Spike Solutions

Supplier	Lot #/ std	Stock
SPEX Certiprep	SPIKE 1-500	**A**
Molybdenum	1000 mg/L	**B**
Ultra	ICUS 192	**C**
Boron	1000 mg/L	**D**
Potassium	10,000 mg/L	**E**

Pre-Digestion Matrix Spike Prep for (GFAA)					
Element	**Spike Amt mL**	**Spike Stock**	**Stock Conc mg/L**	**Final Vol mL**	**Expected Conc. ug/L**
Antimony-GFAA	1.0	**A**	1000.0	50.0	20.0
Arsenic-GFAA	1.0	**A**	1000.0	50.0	20.0
Lead-GFAA	1.0	**A**	1000.0	50.0	20.0
Selenium-GFAA	1.0	**A**	1000.0	50.0	20.0
Thallium-GFAA	1.0	**A**	1000.0	50.0	20.0

Standard Source		
Supplier	**Dilution**	**Solution**
Inorg Vent.-SPK1 (multi-element std)	1/100	**A**

Analyst:		Metals Digestion Summary-CT Laboratories Baraboo								Date:	
Prep Batch#	Sample ID#	Matrix*	Prep Method**	Sample Amt gms or mls	Final Volume, mLs	Blk	Dup	MS	MSD	Spike/LCS/ LFB info	Acid ID#
Method Temperature Verification						*So,Sl,ww,gw,fish,tclp,splp,other				Review date/initials	
Block Used:		Initial Block Temp (C):				**3005,3010,3020,3050,3060				Digestion Tube Lot #:	
Cell #		Final Block Temp (C):				Comments:					

CT Laboratories
Baraboo Laboratory Division

Title: **Acid Digestion of Solids and Semi-solids for Total Metals**

SOP Number: **6230B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare soils, sediments, sludges, oils, paint chips, tissue samples, wipes and other solid waste samples for analysis by ICP, FLAA, or GFAA spectroscopy for the following metals:

Aluminum*	Calcium*	Magnesium*	Silver*
Antimony*	Chromium	Manganese*	Sodium*
Arsenic	Cobalt	Molybdenum	Strontium*
Barium*	Copper*	Nickel *	Thallium
Beryllium	Iron	Potassium*	Vanadium*
Cadmium	Lead	Selenium	Zinc*

* Cannot be used for GFAA for selected element

1.2 For analysis by ICP or FLAA both HCl and HNO_3 are used in the digestion procedure. For analysis by GFAA, HNO_3 only is used.

2.0 METHOD SUMMARY

2.1 For the digestion of samples, a representative 0.5 gram sample or 1-2g sample for ACOE work is digested with repeated additions of nitric acid and hydrogen peroxide

2.2 For GFAA analysis, the resultant digestate is reduced in volume while heating and then diluted to a final volume of 50 mL.

2.3 For ICP (trace) or FLAA analyses, hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. The digestate is then diluted to a final volume of 50 mL.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 Reagent Blank - A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.3 LCS (Laboratory Control Sample)- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the digestion procedure and is analyzed at the beginning of the analytical batch .

3.4 MB (Method Blank)- A reagent blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation process. A minimum of one MB is prepared per batch and is analyzed at the beginning of an analytical batch.

3.5 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every batch (see 3.1) of routine samples. An MS and a DUP are prepared for every ACOE batch. Failure to meet criteria may be due to poor recovery during the preparation method or to matrix interference within the digestate. To be considered acceptable, MSD's must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD may be used as acceptance criteria for duplicate analysis.

3.6 Duplicate (DUP)- A separate aliquot of sample which has been carried through the entire preparation and analytical procedure the same as the original sample. One duplicate per batch is prepared for ACOE work.

4.0 HEALTH AND SAFETY

4.1 Gloves, protective eyewear, and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

5.1 The extracts from GFAA and FLAA/ICP are not interchangeable and should only be used with the analytical determinations outlined.

5.2 This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become “environmentally available.” By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

5.3 Reagent grade chemicals shall be used in all tests. The reagent blank must be less than the MDL in order to be used.

6.0 INTERFERENCES

6.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the

quality control requirements to aid in determining whether Method 3050B is applicable to a given waste.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis shall be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 Personnel shall know how to read a meniscus and how to use a pipette correctly.

8.0 APPARATUS AND MATERIALS

8.1 Equipment Required

8.1.1 Environmental Express Hotblock Digestion Apparatus: To maintain a temperature of 90-95C.

8.1.2 50 mL Class A plastic hotblock digestion tubes, with watch glasses and storage caps. (Environmental Express p/n SC475 and SC505 or equivalent)

8.1.3 Qualitative filter paper and filter funnels.

8.1.4 Filtermate Teflon digestion tube filters: (Environmental Express p/n SC0401 or equivalent)

8.1.5 Thermometer: For monitoring temperature of the block at beginning and end of digestion process.

8.1.6 Balance: 0.01g capacity.

8.2 REAGENTS

8.2.1 ASTM Type II Water (ASTM D1193): Water should be monitored for impurities.

8.2.2 Concentrated Nitric Acid, trace metals grade (HNO_3): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination problems. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with another bottle from the same lot. If the entire lot is a problem, then any remaining acid from the contaminated lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained. All acid used to make standards is recorded in the log book when the standard is made.

8.2.3 Concentrated Hydrochloric Acid, trace metals grade (HCl): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination problems. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with

another bottle from the same lot. If the entire lot is a problem, then any remaining acid from the contaminated lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained. All acid used to make standards is recorded in the log book when the standard is made.

8.2.4 Hydrogen Peroxide, 30% (H_2O_2): Each peroxide lot is logged in and tracked to each individual bottle to identify potential contamination problems. If contamination is detected at any point and the problem is limited to a given bottle, the peroxide may be replaced with another bottle from the same lot. If the entire lot is a problem, then any remaining peroxide from the contaminated lot will be removed from the lab and a replacement hydrogen peroxide bottle from a different lot shall be obtained.

8.2.5 SPEX Certiprep Spike- SPEX catalog number SPIKE 1-500

8.2.6 Molybdenum- 1,000 mg/L standard SPEX PLMO9-2Y or equivalent

8.2.7 Potassium-10,000 mg/L standard PLK2-3X or equivalent

8.2.8 Ultra Custom Standard – Ultra Catalog number ICUS192 or equivalent

8.2.9 Inorganic Venture Spike- Inorganic Venture catalog number SPK1 or equivalent

9.0 INSTRUMENT OR METHOD CALIBRATION

Please refer to the Hot Block operator manual for instrument temperature calibration.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 All sample containers must be demonstrated to be free of contamination at or below the reporting limit. Plastic and glass containers are both suitable. Note: Environmental Express provides a certificate of analysis with each tube lot.

10.2 Samples shall be refrigerated upon receipt and analyzed within 180 days.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Turn on hot blocks to allow them to warm up. The switch is located on the right outer side of hood. Place a thermometer into a centrifuge tube of water and place it in the hot block to monitor the temperature of the hot block to insure the temperature is acceptable prior to use. The temperature should be 95 ± 5^0C for digestion.

11.2 Carefully weigh on a balance and then transfer a 0.5 g representative portion (for routine work) or for ACOE work 1-2g randomly of the sample to a tube, or use a known volume of a sludge sample based on the viscosity of the sample. Do not use metal spatulas. Refer to the subsampling SOP FO-10 for instructions on subsampling from the original sample. For some samples it is acceptable to use a larger sample size following the guidelines below:

Wet solids:	Up to 1.0 g.
Wipes:	The entire wipe is used.
Biological tissue:	Up to 2.0 g.
"liquidy" sludges	Up to 2.0 g

11.3 For samples to be spiked (MS, MSD, LCS), add the appropriate amount of spiking solution to sample (see Pre-digestion Spikes-LCS & LFB chart attached).

11.4 Add 5 mL 1:1 HNO_3 to routine samples or 10mL of 1:1 HNO_3 for ACOE samples, mix, and cover the tube with a watch glass. Do not handle the underside of the watch glass at any time, in order to prevent contamination. Place the tubes in the hotblock and heat the samples until they reflux for 10-15 minutes. For wipe samples, add DI water to wet the entire wipe if needed.

11.5 Add 2.5 mL concentrated HNO_3 to all routine samples and 5mL of concentrated HNO_3 to ACOE samples.

11.6 Place thermometer in one block location, record temperature in the sample prep book for the initial block temperature recording. Record again at the end of the digestion process in a different spot on the block. Rotate the placement of the thermometer between digestion batches to ensure that no "hot spots" develop on the block.

11.7 Return to the hotblock, and reflux the samples for 30 minutes. Do not allow the samples to go dry. If samples are still generating "brown" fumes, continue to add concentrated HNO_3 in 5mL increments until no more "brown" fumes are generated.

11.8 Allow the solution to evaporate to 5 mL without boiling or heat at $95^0C \pm 5^0C$ without boiling for 2 hours. Do not allow samples to go to dryness.

11.9 Remove the samples from the hotblock and let cool to avoid excessive effervescence of hydrogen peroxide. For routine samples add 1 mL of DI water and 1.5 mL of 30% hydrogen peroxide to each tube. For DoD samples add 2 mL of DI water and 3 mL of 30% hydrogen peroxide to each tube (omit this step for Tin). Return to hot block and heat gently until the effervescence subsides.

11.10 Repeat step 11.9 two times or until effervescence is minimal for routine samples using a maximum of 5mL of 30% hydrogen peroxide. For ACOE samples, add 30% hydrogen peroxide in 1mL increments, up to 10mL, until the sample effervescence is minimized or the sample consistency is unchanged. It will not be necessary to cool down the samples between additions if they do not react too strongly.

11.11 Return samples to hot block and heat until volume is reduced to 2.5mL or heat at $95^0C \pm 5^0C$ without boiling for 2 hours.

11.12 For samples to be analyzed by ICP or FLAA, add 5 mL concentrated HCl for routine samples or for ACOE samples add 10mL of concentrated HCl and reflux for an additional 15 minutes. Omit this step for samples to be analyzed by GFAA.

11.13 Remove each tube from block using the tube racks provided and allow them to cool. Dilute to exactly 50 mL using the guide on the digestion tube and let sample sit to allow solids to settle out. If solids do not settle readily and remain suspended throughout sample, then it will be necessary to filter prior to analysis. Using the Environmental Express tube filters, push the filter down through the sample till it sits on the bottom. The digestion tubes are calibrated to 50mL $\pm$ 0.2mL.

11.14 Cap sample, taking care not to touch underside of cap or inside of container. To minimize contamination and for safety purposes appropriate gloves must be worn at any point where sample solution is being transferred. If gloves become stained and dirty, discard and replace, as the surface of the gloves themselves may also transfer contaminants.

11.15 If the digestate is clear and without solids distributed throughout the liquid portion, sample aliquots may be decanted for analysis. If not, then filtration will be necessary prior to analysis. Follow instruction provided with filter-mate filters.

12.0 TROUBLESHOOTING

12.1 If method blank is higher than MDL, identify source of contamination. Contamination can come from many sources including the reagents used in the procedure and samples that were digested in the batch.

12.2 Over time, hot blocks can develop “hot spots”. Monitor the temperature in different block spaces ensure that temperature is consistent throughout the hot block unit.

13.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

There are no calculations for this method.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Batch data information is recorded in the Digestion Log book.

15.2 Prep data is entered into LIMS, and then the batch sheet is given to the metals analyst.

16.0 QUALITY CONTROL

16.1 For each batch of samples processed, a method blank should be carried throughout the entire sample preparation procedure.

16.2 By matrix, prepare one MS/MSD for routine work or one MS/DUP for ACOE work per 20 or fewer samples per matrix per digestion batch.

16.3 Prepare one LCS (lab control sample) for each digestion batch. The LCS source is carried through the entire digestion procedure. Prepare one LCS for every 20 samples per medium per day.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*. USEPA SW-846, Rev 2 Dec 1996, Method 3050B.

17.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, January, 2006.

18.0 Prep Tables

18.1 ICP/FLAA Table

SPIKE, & LFB ANALYSIS- ICP/FLAA Predigestion Spikes-LCS & LFB					
Element	**Spike Amt mL**	**Spike Stock**	**Stock Conc mg/L**	**Final Vol mL**	**Expected Conc. ug/L**
Aluminum	1	**A**	200	50	4000
Antimony	1	**A**	50	50	1000
Arsenic	1	**A**	200	50	4000
Barium	1	**A**	200	50	4000
Beryllium	1	**A**	5	50	100
Cadmium	1	**A**	5	50	100
Calcium	0.5	**C**	20,000	50	200000
Chromium	1	**A**	20	50	400
Cobalt	1	**A**	50	50	1000
Copper	1	**A**	25	50	500
Iron	1	**A**	100	50	2000
Lead	1	**A**	50	50	1000
Manganese	1	**A**	50	50	1000
Magnesium	0.5	**C**	10,000	50	100000
Molybdenum	0.1	**B**	1000	50	2000
Nickel	1	**A**	50	50	1000
Potassium	0.5	**D**	10,000	50	100000
Selenium	1	**A**	200	50	4000
Silver	1	**A**	5	50	100
Sodium	0.5	**C**	10,000	50	100000
Thallium	1	**A**	200	50	4000
Vanadium	1	**A**	50	50	1000
Zinc	1	**A**	50	50	1000

Spike Solutions		
Supplier	**Lot #/ std**	**Stock**
SPEX Certiprep	SPIKE 1-500	**A**
Molybdenum	1000 mg/L	**B**
Ultra	ICUS 192	**C**
Potassium	10,000	**D**

18.2 GFAA Table

Pre-Digestion Matrix Spike Prep for (GFAA)					
Element	**Spike Amt mL**	**Spike Stock**	**Stock Conc mg/L**	**Final Vol mL**	**Expected Conc. ug/L**
Antimony-GFAA	1.0	**A**	1000.0	50.0	20.0
Arsenic-GFAA	1.0	**A**	1000.0	50.0	20.0
Lead-GFAA	1.0	**A**	1000.0	50.0	20.0
Selenium-GFAA	1.0	**A**	1000.0	50.0	20.0
Thallium-GFAA	1.0	**A**	1000.0	50.0	20.0

Standard Source		
Supplier	**Dilution**	**Solution**
Inorg Vent.-SPK1 (multi-element std)	1/100	**A**

Analyst:		Metals Digestion Summary-CT Laboratories Baraboo								Date:	
Prep Batch#	**Sample ID#**	**Matrix***	**Prep Method****	**Sample Amt gms or mls**	**Final Volume, mLs**	**Blk**	**Dup**	**MS**	**MSD**	**Spike/LCS/ LFB info**	**Acid ID#**

Method Temperature Verification				*So,Sl,ww,gw,fish,tclp,splp,other	Review date/initials	
Block Used:		Initial Block Temp (C):		**3005,3010,3020,3050,3060	Digestion Tube Lot #:	
Cell #		Final Block Temp (C):		Comments:		

CT Laboratories
Baraboo Laboratory Division

Title: **Total Metals Sample Preparation for FLAA and ICP**

SOP Number: **6225B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, by ICP or FLAA, for the metals listed below. The procedure is used to determine total metals.

1.2 Samples prepared by this method may be analyzed by ICP or FLAA for the following:

Aluminum	Calcium	Lead
Potassium	Arsenic*	Chromium
Magnesium	Selenium*	Barium
Cobalt	Manganese	Sodium
Beryllium	Copper	Molybdenum
Thallium	Cadmium	Iron
Nickel	Vanadium	Zinc
Boron*		

*Analysis by ICP

1.3 This digestion procedure is not suitable for samples to be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interferences during furnace atomization.

2.0 METHOD SUMMARY

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered digestion tube. This step is repeated with additional portions of nitric acid. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 Reagent Blank - A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.3 LCS (Laboratory Control Sample)- A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the digestion process. A minimum of one LCS is prepared per batch and is analyzed at the beginning of an analytical batch.

3.4 PB or MB (Prep Blank or Method Blank)- A reagent blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation process. A minimum of one MB is prepared per batch and is analyzed at the beginning of an analytical batch.

3.5 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix

spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every batch of routine samples or an MS/DUP pair is prepared for every batch of ACOE samples. Failure to meet criteria may be due to poor recovery during the preparation method or to matrix interference within the digestate. To be considered acceptable, MSD's must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD and may be used as acceptance criteria for duplicate analysis.

3.6 Detection Limit (DL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

3.7 Duplicate (DUP)- A separate aliquot of sample which has been carried through the entire preparation and analytical procedure the same as the original sample. One duplicate per batch is prepared for ACOE work.

4.0 HEALTH AND SAFETY

4.1 Gloves, protective eyewear, and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems

5.0 CAUTIONS

5.1 Do not allow samples to evaporate to dryness. If the samples do go to dryness, they must be reprepped.

5.2 Use caution when adding acid to hot samples. Sample can foam and splash when the acid is being added.

6.0 INTERFERENCES

6.1 This digestion procedure is not suitable for samples to be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interferences during furnace atomization.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis should be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 All personnel performing this analysis should have a valid IDC prior to performing preparation on samples.

8.0 APPARATUS AND MATERIALS

8.1 Environmental Express Hotblock Digestion Apparatus: To maintain a temperature of 90-95C.

8.2 50 mL Class A Graduated hotblock digestion tubes, with watch glasses and storage caps (Environmental Express p/n SC475 and SC505 or equivalent)

8.3 Qualitative filter paper and filter funnels.

8.4 Filtermate Teflon digestion tube filters: (Environmental Express p/n SC0401 or equivalent)

8.5 Thermometer: For monitoring temperature of the block at beginning and end of digestion process.

8.6 Acrylic digestion tube carriers. For suspending tubes in blocks and for cooling tubes on the counter.

8.7 Foam digestion tube racks for storage of prepped samples.

8.8 REAGENTS

8.8.1 ASTM Type II Water (ASTM D1193): Water should be monitored for impurities.

8.8.2 Concentrated Nitric Acid, trace metals grade (HNO_3): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with another bottle from the same lot. If the entire lot is contaminated, then any remaining acid from the lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained. All acid used to make standards is recorded in the log book when the standard is made.

8.8.3 Concentrated Hydrochloric Acid, trace metals grade (HCl): Acid is of sufficient purity and comes with a certificate of analysis. All metals levels are lower than our lowest detection limits for any given element. However, each acid lot is logged in and tracked to each individual bottle to identify potential contamination. If contamination is detected at any point and the problem is limited to a given bottle, the acid may be replaced with another bottle from the same lot. If the entire lot is contaminated, then any remaining acid from the lot will be removed from the lab and a replacement acid from a different lot of sufficient purity shall be obtained. All acid used to make standards is recorded in the log book when the standard is made.

8.8.4 Spike Standard Solution- SPEX cat # SPIKE-1-500

8.8.5 Molybdenum Standard- 1000 mg/L

8.8.6 Custom Standard- Ultra # ICUS 192- 20,000 mg/L Ca, 10,000 mg/L Mg and Na or equivalent

8.8.7 Potassium standard- 10,000 mg/L

8.8.8 Boron standard- 1,000 mg/L

8.8.9 Lithium standard- 1000 mg/L

9.0 INSTRUMENT OR METHOD CALIBRATION

9.1 There is no calibration necessary

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

	Total Metals
Preservative	pH <2 HNO_3
Hold Time	180 days

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Turn on hot blocks by turning the silver timer switches, located on the right side of hood, clockwise. Allow hot blocks to come to temperature before placing samples into the blocks. Monitor temperature of the hot block by placing a thermometer immersed in water in a centrifuge tube and place it in the hot block. Temperature should be 90-95^{0}C for digestion. Move thermometer to various places on the block between digestion batches to insure that the the temperature is consistent throughout the block and that no "hot spots" have developed.

11.2 Measure 50 mls of well-mixed sample into a clean, labeled hot-block digestion tube. For blanks use same amount of Type II water.

11.3 For samples to be spiked (MS, MSD, LCS), add the appropriate amount of spiking solution to sample (see Predigestion Spikes-LCS & LFB chart attached).

11.4 Add 1.5 mL conc. Trace Metal Grade HNO3 from dispenser.

11.5 Cover with a plastic ribbed watch-glass for samples that may react strongly to acid or heat. Lower the tube racks into hot-block and heat at 90-95^0 C until the volume has been reduced to about 3 mL. Do not allow sample to go dry. If this should occur, discard the sample and start over.

11.6 Remove tubes from block using rack. Add 1.5 mL conc. HNO3. Cover all tubes with watch-glasses. Place in block and reflux for one hour. Do not allow to go to dry. Continue addition of acid and heating until digestion is complete (usually indicated when the digestate is light colored or does not change in appearance while refluxing. Most samples will not require additional acid and heat cycles).

11.7 Remove tubes from block using rack. Add 5 mL of 1:1 Trace Metal Grade HCl. Cover tubes, and reflux for 15 minutes.

11.8 Remove each tube from block using the tube racks provided and allow them to cool.

11.9 Dilute to original volume inside tube and let sample sit to allow solids to settle out. . Hot block tubes are calibrated to volume $\pm$ 0.2mL. If solids do not settle readily and remain suspended throughout sample, then it will be necessary to filter prior to analysis. Using the Environmental Express filter-mates, push the filter down through the sample until it sits on the bottom. Alternately, filter through acid rinsed filter paper, rinsing at least 3 times into the funnel until filled to the 50 mL line. When using the tube filter fill tube to the 50 mL line first. If using filter and funnel, dilute to 50 mL line after.

11.10 Cap sample, taking care not to touch underside of cap or inside of container. To minimize contamination and for safety purposes appropriate gloves must be worn at any point where sample solution is being transferred. If gloves become stained and dirty, discard and replace as the surface of the gloves themselves may also transfer contaminants.

12.0 TROUBLESHOOTING AND MAINTENANCE

12.1 Refer to the Environmental Express manual for troubleshooting directions.

13.0 DATA ACQUISITION, CALCULATIONS AND REDUCTION

13.1 There are no calculations associated with this procedure.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS.

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Data is recorded in the Metals digestion log.

15.2 Prep data is entered into LIMS, and then the batch sheet (complete with standards and acids reference #'s) is given to the metals prep analyst.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 For each analytical batch of samples processed, a method blank (Type II water and reagents) should be carried throughout the entire sample preparation procedure.

16.2 Prepare one Matrix Spike (MS) and one Matrix Spike Duplicate (MSD) per matrix per 20 or fewer samples per digestion batch. MS/MSD's shall include all targeted analytes spiked into the parent sample..

16.3 Prepare one LCS (lab control sample) for each digestion batch by spiking reagent water with the same concentration of analyte(s) used for the MS/MSD. The LCS is prepared from one or more commercially prepared reference standards but must be from a source different from that used to prepare the calibration standards for a given instrument. The LCS solution is carried through the entire digestion procedure. Prepare one LCS for every 20 samples on a given matrix per day.

16.4 Monitor block thermometer in one block location, record temperature in the sample prep book after 30 min, for the initial block temperature recording. Record again at the end of the digestion process.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste. EPA, SW-846, 3rd Edition, 1992 Method 3010A.

17.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 3, January, 2006.

SPIKE, LCS, & LFB ANALYSIS- ICP/FLAA Predigestion Spikes-LCS & LFB					
Element	**Spike Amt mL**	**Spike Stock**	**Stock Conc mg/L**	**Final Vol mL**	**Expected Conc. ug/L**
Aluminum	1	**A**	200	50	4000
Antimony	1	**A**	50	50	1000
Arsenic	1	**A**	200	50	4000
Barium	1	**A**	200	50	4000
Beryllium	1	**A**	5	50	100
Boron	0.1	**D**	1,000	50	2000
Cadmium	1	**A**	5	50	100
Calcium	0.5	**C**	20,000	50	200000
Chromium	1	**A**	20	50	400
Cobalt	1	**A**	50	50	1000
Copper	1	**A**	25	50	500
Iron	1	**A**	100	50	2000
Lead	1	**A**	50	50	1000
Manganese	1	**A**	50	50	1000
Magnesium	0.5	**C**	10,000	50	100000
Molybdenum	0.1	**B**	1000	50	2000
Potassium	0.5	**E**	10,000	50	100000
Nickel	1	**A**	50	50	1000
Selenium	1	**A**	200	50	4000
Silver	1	**A**	5	50	100
Sodium	0.5	**C**	10,000	50	10,000
Thallium	1	**A**	200	50	4000
Vanadium	1	**A**	50	50	1000
Zinc	1	**A**	50	50	1000

Spike Solutions		
Supplier	**Lot #/ std**	**Stock**
SPEX Certiprep	SPIKE 1-500	**A**
Molybdenum	1000 mg/L	**B**
Ultra	ICUS 192	**C**
Boron	1000 mg/L	**D**
Potassium	10,000 mg/L	**E**

Analyst:_____ **Metals Digestion Summary - CT Laboratories Baraboo, WI** **Date:_____**

Prep Batch#	Sample ID#	Matrix*	Prep Method**	Sample Amt (g or ml)	Final Vol. (ml)	Blk	Dup	MS	MSD	Spike/LCS/LFB Log ID #(s)	Acid Log ID#

Method Temperature Verification		*SO,SL,WW,GW,TCLP,SPLP,ASTM, other **3005,3010,3020,3050,3060		Review Date/Initials:	
Block Used:		Initial Block Temp (C):		Digestion Tube Lot #:	
Cell #		Final Block Temp (C):		Comments:	

CT Laboratories
Baraboo Laboratory Divison

Title: Reactive Cyanide Distillation

SOP Number: CC-RCN-Distillation

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance *Date*

_____
Laboratory Director *Date*

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method covers the distillation of reactive cyanide in wastes provided that the addition of acid to the waste does not form an explosive mixture.

1.2 The distillate collected from this procedure is analyzed on the Lachat according the method CC-1.

2.0 METHOD SUMMARY

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry (CC-1).

3.0 DEFINITIONS

3.1 Lab Control Sample (LCS): A known concentration obtained from a source different than the calibration.

3.2 Matrix spike-matrix spike duplicate (MS-MSD): To two of three aliquots of a given sample a known amount of spike solution is added. Matrix spikes and matrix spike duplicates are taken through all steps of preparation and analysis, exactly like a sample. The amount of spike recovered helps to assess the effect of the sample matrix on the analysis. The precision of the method is also determined, by calculating the relative percent difference (RPD) (10.3) of the two spiked aliquots. A MS-MSD should be prepared for every 20 samples in each matrix.

3.3 Method Blank (MB): Reagent water treated exactly as a sample, including exposure to all glassware, reagents, and treatments used with other samples.

3.4 Batch- A batch consists of 20 samples of the same matrix analyzed on the same day or 20 samples of the same medium that have been prepared together.

4.0 HEALTH AND SAFETY

4.1 Cyanogen chloride may form during this reaction. This is a very toxic gas and the distillation should always be performed in a hood.

4.2 Caution should be used during the distillation step. If the vacuum pressure is not adequately maintained, sulfuric acid or sodium hydroxide could be expelled from the distillation apparatus.

4.3 Caution: KCN is highly toxic. Avoid inhalation of dust or contact with the solid or solutions.

5.0 CAUTIONS

5.1 A minimum of 50mL or 5.0g of sample is required for analysis. Samples must be preserved with sodium hydroxide to pH of >12 , and cooled to 4 ^{o} C at the time of collection (approximately 2 mL NaOH/1-L sample). Soil samples can be taken in unpreserved 4 oz. jars and cooled to 4º C until analysis.

5.2 Samples may not be stored for longer than 14 days.

6.0 INTERFERENCES

6.1 Chlorine: Oxidizing agents, such as chlorine, decompose most of the cyanides. To determine if there is significant chlorine present, test a drop of the sample with potassium-iodide starch paper. If the paper turns blue, treatment with ascorbic acid will be necessary prior to analysis. Add a few crystals of ascorbic acid a time, until a drop of sample produces no color on the indicator paper; then add an additional 0.6 g of ascorbic acid for each liter of sample.

6.2 Sulfides: Test for presence of sulfide by placing a drop of sample on lead acetate test paper. Darkening of paper indicates presence of sulfide. Treat 75 mL of the reserved sample (pH>12) as follows: Add powdered Cadmium Carbonate to sample gradually. Yellow cadmium sulfide precipitates if the sample contains sulfide. Test the treated sample periodically by adding a drop of the solution to the lead acetate paper. When the paper does not darken, the sulfide has been removed sufficiently. Filter the treated solution through a dry filter paper into a dry beaker. From this filtrate measure out the sample aliquot to be used for analysis. Note any treatment in the sample prep logbook. Avoid a large excess of cadmium and a long contact time.

6.3 NO_3/NO_2: Remove by adding excess (0.2 g) sulfamic acid before distillation.

6.4 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic acid (1.6M) to pH 6.0 to 7.0. **CAUTION: This procedure can produce lethal HCN gas.** Extract with hexane with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of thesample to above 12 with 50% NaOH solution.

6.5 Most interferences are eliminated reduced by the distillation procedure.

7.0 PERSONNEL QUALIFICATIONS

7.1 Analysts should be familiar with safety requirements for the lab.

7.2 Analysts should have a valid IDC on file.

8.0 APPARATUS AND MATERIALS

8.1 Materials

8.1.1 Fisher Analytical Balance: To weigh accurately to the nearest 0.0001 g.
8.1.2 Glassware: Class A volumetric flasks and pipets.
8.1.3 Midi-dist Apparatus: Easy-Dist model, with vacuum controls, digestion tubes, and gas scrubbers.
8.1.4 Twistcap plastic centrifuge tubes. Stored in wetchem lab.

8.2 Reagents
8.2.1 All reagents are prepared with deionized water.
8.2.2 Stock Cyanide Solution (1000 mg CN/L): Puchased commercially, Ultra Scientific-Cat. # ICC-008. This standard is used for CCVs and calibrating the Lachat (See method CC-1). Second Source = Labchem-Cat. # LC13545-7. This standard will be used for the IVC and spiking. Alternatively prepare the stock standard by dissolving 2.5g KOH and 2.51g KCN in 1L reagent water. Standardize before use (See method CC-1).
8.2.3 . 0.25M NaOH: In a 1L volumetric flask dissolve 10g NaOH and bring to volume with reagent water.
8.2.4 LCSW (2mg/L): Add 0.1mL of 1000mg/L CN Stock solution (8.2.2) to 50mL 0.25M NaOH (8.2.3)
8.2.5 LCSS (20 mg/Kg): Add 0.1mL of 1000 mg/L CN- stock solution (8.2.2) and 50mL 0.25NaOH (8.2.3) to 5.0g sand.
8.2.6 Sulfuric Acid 0.5N: Add 14mL of concentrated H2SO4 (stored under hood in glassware room) to 1000mL of reagent water in 1L volumetric while under a hood. Cool and bring to volume with reagent water.
8.2.7 Standardized Silver Nitrate Solution, 0.0192 N: NIST traceable. (stored under the hood in the extraction lab.)
8.2.8 Indicator solution: 0.02% p-dimethylaminobenzalrhodanine w/v in acetone.

9.0 INSTRUMENT OR METHOD CALIBRATION

See Method CC-1.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 A minimum of 50 mL of sample is required for liquid analysis. Samples must be preserved with sodium hydroxide to pH of >12 , and cooled to 4 ^{o} C at the time of collection (approximately 2 mL NaOH/1-L sample). Soil samples can be taken in unpreserved 4 oz. jars and cooled to 4º C until analysis.
10.2 Samples may not be stored for longer than 14 days.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Pour 50 mL 0.25 M NaOH into each scrubber tube.
11.2 For liquid samples, add 50 mL of sample or an aliquot diluted to 50 mL to the midi-distillation boiling tube. For soil samples: weigh up

approximately 5 g of soil and place in boiling tube, then add 50 mLs of 0.25 NaOH.

11.3 Assemble each apparatus. Start the vacuum after all connections are made and checked. Adjust the vacuum bubble rate at approximately 2 bubbles/ second. If you are distilling a soil the bubble rate should be higher to ensure adequate mixing of reagent with the soil in suspension. Let bubbles mix the solution for about 3 minutes. For spiked samples, add 0.1 mL of 1000mg/L cyanide standard with a calibrated pipettor through the air inlet at this point. Rinse down with a small amount of reagent water, then let the solution mix for 3 minutes.

11.4 Slowly add 5 mL of 0.5N H_2SO_4 through the air inlet tube. Rinse down with a small amount of reagent water, continue to bubble 3 minutes.

11.5 Do **NOT** turn on the Easy-Dist. Let the acidified samples bubble for at least 30 minutes.

11.6 After distillation finishes, shut off the vacuum and remove the absorber tube.

11.7 Pour off the trapped cyanide solutions into 50 mL twistcap plastic centrifuge tubes. Prepped samples can be stored for later analysis for up to 3 days. Keep refrigerated at 4^oC in the walk-in cooler until analysis.

11.8 Samples are analyzed according to method CC-1. The true value of the LCS and spiked samples are initially over-range and should be diluted x20 before analysis.

12.0 TROUBLESHOOTING AND MAINTENANCE

12.1 Ensure that the LCS is free cyanide. This method will not recover cyanide complexes.

12.2 Cyanide breaks down over time particularly in the presence of light. If LCS recovery is low reprepare the spiking solution and/or standardize the prepared stock standard to ensure the correct values.

12.3 If the MB fails determine the source of contamination. Eliminate and redistill.

13.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

Liquid Concentration (mg/L) = A x C x 1000

$$\textit{Solid Concentrations (mg/kg)} = \frac{A \times B \times C}{D \times E},$$

where A = instrument reading for sample (ug/L)
B = total volume of digestion (L)
C= analyst dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary

$$\textit{Spike Recovery (\%)} = \frac{(\text{Spiked sample concentration} - \text{Sample concentration}) \times 100}{}$$

$$\%RSD = \frac{(MS - MSD) \times 100}{(MS + MSD)/2 \quad \text{(Spike amount)}}$$

where MS = Matrix spike concentration
MSD = Matrix spike duplicate concentration

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS

14.2 Lachat QuickChem 8000 software

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.

15.2 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

15.3 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.

15.4 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 The analyst must show an initial demonstration of capability (IDC) to generated acceptable data, by successfully analyzing four replicates of a known standard and having acceptable results for a blind sample.

16.2 ICV (Initial Calibration Verification): This mid range check standard is analyzed after calibration. Control limits are +/- 10% of the true value. If the recovery is outside of this range, terminate the run and correct the problem before proceeding.

16.3 ICB (Initial Calibration Blank): Analyzed immediately after the ICV. The value of the ICB must be below the method LOD. If not, terminate the analysis and correct the problem before proceeding or qualify results less than 20 times the ICB with a 'B' flag.

16.4 LCS (Laboratory Control Sample): Analyze a LCS (reference obtained from a second source) per 20 samples. Control limits are +/- 50% of the true value. An out of control LCS may indicate that the calibration standard stock solution is inaccurate or that there was a problem with the distillation process. If the recovery exceeds this, terminate the analysis and correct the problem before proceeding

16.5 CCV (Continuing Calibration Verification): Analyze a CCV following every ten samples and at the end of the analysis. Control limits are +/- 10% of the true value. If recovery is outside these limits, recalibrate and reanalyze all samples back to the last acceptable CCV or ICV.

16.6 CCB (Continuing Calibration Blank): Analyzed immediately after the CCV. The value of the CCB must be below the method LOD. If not,

terminate the analysis and correct the problem before proceeding. If CCB is outside these limits, recalibrate and reanalyze all samples back to the last acceptable CCB or ICB or qualify results less than 20 times the CCB with a 'B' flag.

16.7 MS-MSD (Matrix Spike-Matrix Spike Duplicate):A MS-MSD is required every analytical run at a frequency of 5% (1 for every 20 samples) per matrix type. Refer to the quarterly control limits for acceptance criteria for both accuracy and precision. If recovery is outside of acceptable range, flag the sample with an 'M' qualifer. If the RPD is outside of the acceptable range, flag the sample with a 'Y' qualifier.

16.8 MB (Method blank): analyze one method blank per digestion batch. This is a reagent water blank that is taken through all of the processes that the samples are. The value of the method blank must be below the method LOD. If not, terminate the analysis and correct the problem before proceeding or qualify results less than 20 times the method blank with a 'B' flag.

16.9 If the 1000 mg/L CN stock standard is prepared in the lab then standardized against a 0.0192N $AgNO_3$ solution biweekly. Acceptance criteria, is +/-0.5% CN.

17.0 REFERENCES

17.1 Chapter 7, USEPA, SW-846, Rev. 2, Sept. 1994

17.1 *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA-600/R-93/ 100, August 1993, Method 335.4.

17.2 *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 1992, APHA-AWWA-WEF, Method 4500-CN.

17.3 *SW 846*, 3rd Edition, Update III, 1997 Method 9010B, 9012A and 9013.

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Six-point initial calibration plus blank (ICAL)	Initially and as needed	r $\geq$ 0.995 for regression line	Repeat until acceptable
Initial calibration verification (ICV)	After each ICAL, prior to sample analysis	%R: 90-110% for all analytes	Remake and reanalyze ICV standard, if still unacceptable repeat ICAL
Initial calibration blank (ICB)	After each ICV, prior to sample analysis	Program Specific or < RL	Remake and reanalyze CB once, if still unacceptable investigate and correct problem or flag results less than 20 X's the ICB with a 'B' qualifier
Continuing calibration verification (CCV)	Daily, prior to sample analysis, after every 10 samples, and at end of run	%R: 90-110% for all analytes	Remake and reanalyze CCV, if still unacceptable investigate and correct problem. Reanalyze all samples after last acceptable CCV.
Continuing calibration blank (CCB)	After each CCV	Program Specific or <RL or <20 times less than the lowest sample result	Remake and reanalyze CB once, if still unacceptable investigate and correct problem then reanalyze all samples after the last acceptable CCB or flag results less than 20 X's the CCB with a 'B' qualifier
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses and one blind sample	In-house determined criteria for LCS recovery and precision	Repeat until acceptable
Method Blank (MB)	One (1) per analytical batch	Program Specific or <RL or <20 times less than the lowest sample result	Clean analytical system, repeat. If still unacceptable, redistill batch or flag all results less than 20 X's the MB with a 'B' qualifier.
Matrix Spike (MS)	One (1) per analytical batch per matrix	Program specific or In-house derived limits	Investigate problem, if LCS in control qualify results. Qualify results with a 'M' flag if necessary
Matrix Spike Duplicate (MSD)	One (1) per analytical batch per matrix	Program specific or In-house derived limits	Investigate problem, if LCS in control qualify results. Qualify results with a 'M' or 'Y' flag if necessary
Sample duplicate (QSM/LCG))	One (1) per analytical batch per matrix	+/- 20 % Diff.	Investigate problem, if non apparent qualify results. Qualify results with a 'Y' flag if necessary
Low and High Distillation Standards (QSM/LCG)	One (1) per analytical batch	Within +/- 15% of true value	Troubleshoot and reanalyze. If fails again redistill samples
Laboratory control sample (LCS)	One per analytical batch	Program specific or In-house derived limits	Halt analysis, fix problem, repeat associated sample analyses. If still unacceptable, redistill batch

Data Validation Checklist

LIMS #:	Method: REACTIVE CYANIDE	

Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a distillation sheet accompany the data package?	---					
2. Does the distillation sheet include all reagent and standard prep log numbers?	---					
3. Is a copy of the stock standard standardization log included?	---					
4. Is the stock standard within acceptance limits?	+/- 0.5% of reported value					
5. Were samples checked for chlorine and sulfide?	---					
6. Were the samples distilled within hold time?	14 days					
7. Were method blanks prepared at the required frequency?	1per 20 samples					
8. Was the method blank result acceptable?	< LOD					
9. Were LCS's prepared at the required frequency?	1per 20 samples					
10. Was the LCS used before the indicated expiration date?	---					
11. Was the LCS recovery acceptable?	Program specific or within in-house QC limits					
12. Were the MS and MSD prepared at the required frequency?	1per 20 samples of the same matrix					
13. Were the MS and MSD recoveries acceptable?	Program specific or within in-house QC limits					
14. Was the RPD between the MS and MSD acceptable?	Program specific or within in-house QC limits					

Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
15. Was calibration performed using required number of standards	6 plus a blank					
16. Is the standard prep log number noted on the analytical report?	---					
17. Was the correlation coefficient acceptable?	>= 0.995					
18. Were distilled calibration standard recoveries acceptable?	90-110					
19. Were the ICV and ICB run immediately after the calibration curve?	---					
20. Was the ICV recovery acceptable?	90 – 110 %					
21. Was the ICB result acceptable?	< LOD					
22. Were the CCV's and the CCB's analyzed at the required frequency?	1per 10 samples					
23. Were the CCV recoveries acceptable?	90 – 110 %					
24. Were the CCB results acceptable?	< LOD					
25. Were the samples analyzed within 3 days of being distilled?	---					
26. Was a sample run in Duplicate and was precision acceptable (QSM/LCG data)?	+/- 20 % Diff.					
27. Were distilled High/Low standard recoveries acceptable (QSM/LCG)?	85 – 115 % Rec.					
28. Are all samples on the job lists accounted for?	---					

CT Laboratories
Baraboo Laboratory Division

Title: Reactive Cyanide Pre-Test

SOP Number: CC-RCN-Screen

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance *Date*

_____
Laboratory Director *Date*

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 This method covers the qualitative determination of reactive cyanide in wastes. If samples test positive they must then be quantitatively tested according to the reactive cyanide method.

1.2 This method does not cause the reduction of the many cyanide complexes. Therefore it **cannot** be used for a qualitative determination of total cyanide.

2.0 METHOD SUMMARY

2.1 The reactive cyanide, as hydrogen cyanide gas (HCN), is released from samples containing cyanide by acidifying the sample to pH <2.0. The presence of hydrogen cyanide gas is indicated by a color change in the cyanide screening paper held just above the solution.

3.0 DEFINITIONS

3.1 Reactive cyanide refers to cyanide in such a form that it will produce toxic gas (hydrogen cyanide) when exposed to pH conditions between 2 and 12.5.

3.2 Lab Control Sample (LCS): A standard of a known concentration of free cyanide, analyzed at a rate of one per batch of samples.

3.3 Matrix spike-matrix spike duplicate (MS-MSD), when requested: To two of three aliquots of a given sample a known amount of spike solution is added. Matrix spikes and matrix spike duplicates are taken through all steps of preparation and analysis, exactly like a sample. Spike recovery helps to assess the effect of the sample matrix on the analysis. An MS-MSD should be C in each matrix.

3.4 Method Blank (MB): Reagent water treated exactly as a sample, including exposure to all glassware, reagents, and treatments used with other samples; analyzed at a rate of one per batch of samples.

3.5 Batch- A batch consists of 20 samples (or fewer) of the same matrix analyzed on the same day or 20 samples of the same medium that have been prepared together.

4.0 HEALTH AND SAFETY

4.1 Hydrogen Cyanide may form during this reaction. This is a very toxic gas and the analysis should always be performed in a hood.

4.2 Caution: KCN is highly toxic. Avoid inhalation of dust or contact with the solid or solutions.

5.0 CAUTIONS

5.1 Samples should be analyzed so soon as possible. They may not be stored for longer than 14 days.

6.0 INTERFERENCES

6.1 Strong oxidizers may interfere by darkening the test paper. Interference is eliminated during the quantitative analysis of samples with positive test results.

7.0 PERSONNEL QUALIFICATIONS

7.1 Analysts should be familiar with safety requirements for the lab.

8.0 APPARATUS AND MATERIALS

8.1 Materials

8.1.1 Fisher Analytical Balance: To weigh accurately to the nearest 0.0001 g.

8.1.2 Specimen cups and caps

8.1.3 Cyantesmo test paper

8.1.4 Range 0-14 pH paper

8.2 Reagents

8.2.1 All reagents are prepared with deionized water.

8.2.2 Stock Cyanide Solution (1000 mg/L, Ultra Sci- Cat. # ICC-008 or equivalent): . This standard will be used for spiking.

8.2.3 1 N NaOH: In a 1L volumetric flask dissolve 40g NaOH and bring to volume with reagent water.

8.2.4 LCSW (10mg/L): Add 0.1mL of 1000mg/L CN Stock solution (8.2.2) to 10mL 1N NaOH (8.2.3)

8.2.5 LCSS (20 mg/Kg): Add 0.1mL of 1000 mg/L CN- stock solution (8.2.2) to10ml 1N NaOH and 5.0g sand

8.2.6 Sulfuric Acid 6N: Slowly add 196mL of concentrated H2SO4 (stored under hood in glassware room) to 500mL of reagent water in 1L volumetric while under a hood. Cool and bring to volume with reagent water.

9.0 INSTRUMENT OR METHOD CALIBRATION

9.1 The method is confirmed by a positive LCS result. Negative results are reported less than the LCS value. Positive results must be quantified according to the reactive cyanide method .

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 A minimum of 10mL or 5g of sample is required for analysis. H2O samples must be preserved with sodium hydroxide to pH of >12 , and cooled to 4 ^{o} C at the time of collection (approximately 2mL NaOH/1-L sample). Soil samples can be taken in unpreserved 4 oz. jars and cooled to 4° C until analysis.

10.2 Samples may not be stored for longer than 14 days.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 Place 5.0 grams or 10 mL of sample in a specimen cup.

11.2 Place a strip of cyantesmo test paper in the cup so that it extends from the bottom to the top bottom of the container.

11.3 Acidify the sample with 5mL of 6M H2SO4. Immediately cap the solution.
11.4 Gently mix the solution taking care not to completely emerse the test paper (the paper will not change color where the sample has touched it).
11.5 After 30 minutes examine the test paper for a color change. The presence of cyanide is indicated by a blue color beginning at the edge of the paper.
11.6 Check the pH of the sample. If the pH is greater than two, repeat the analysis adding enough acid to lower to pH to less than two.

12.0 TROUBLESHOOTING AND MAINTENANCE

12.1 If the LCS does not test positive for cyanide, remake it (ensuring that it is made with free cyanide rather than cyanide complexes).
12.2 If the MB tests positive, retest. If it still tests positive use new test paper.

13.0 DATA ACQUISITION, CALCULATIONS AND DATA REDUCTION

RANDY ENTER CALCS HERE

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 For work that requires reactive CN screening results are entered as positive or negative. For true reactive cyanide testing negative results are entered as less than the LCS value. Positive results must be quantitatively tested according to the reactive cyanide SOP (CC-RCN-Distillation)
15.2 After data has been entered in LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist for data review guidance.
15.3 Once analyst has processed and enters the data, it is given to a peer or supervisor for reveiw.
15.4 After the second reviewer approves the data, they designate it to "validated" status in LIMS.
15.5 The original data is filed by test in the file cabinet and periodically the contents of the file cabinet are archived.

16.0 QUALITY CONTROL AND QUALITY ASSURANCE

16.1 LCS (lab control sample): Analyze a per 20 samples. LCS must have a positive result.
16.2 MS (matrix spike):A MS should be analyzed with every analytical run at a frequency of 5% (1 for every 20 samples) per matrix type. If there is **not** a positive result, test according to the reactive cyanide SOP(CC-RCN-Distillation)
16.3 Method blank: analyze one method blank per digestion batch. This is a reagent water blank that is taken through all of the processes that the samples are. The method blank must test negative. If not, terminate the analysis and correct the problem before proceeding.

17.0 REFERENCES

17.1 *ASTM D5049-90, Standard Test Method for the Screening of Cyanides in Waste.*

SUMMARY OF QUALITY CONTROL REQUIREMENTS

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Method Blank (MB)	One (1) per analytical batch	Negative Result	Clean glassware, repeat. If still unacceptable replace test paper.
Matrix spike sample (MS)	One (1) per analytical batch per matrix	Positive Result	test according to the reactive cyanide SOP
Laboratory control sample (LCS)	One per analytical batch	Positive Result	Halt analysis, fix problem, repeat associated sample analyses. If still unacceptable, redistill batch.

Data Validation Checklist

LIMS #:	Method: CYANIDE SCREENING			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a bench sheet accompany the data package?	---					
2. Does the bench sheet include all reagent numbers?	---					
3. Were the samples analyzed within hold time?	14 days					
4. Were method blanks prepared at the required frequency?	1per 20 samples					
5. Was the method blank result acceptable?	Negative					
6. Were LCSs prepared at the required frequency?	1per 20 samples					
7. Was the LCS used before the indicated expiration date?	---					
8. Was the LCS recovery acceptable?	Positive					
9. Was a MS prepared at the required frequency?	1per 20 samples of the same matrix					
10. Was the MS recovery acceptable?	Positive					
11. Are all samples on the job lists accounted for?	---					

CT Laboratories
Baraboo Laboratory Division

Title: **Analysis of Semivolatile Organic Compounds by GC/MS 8270**

SOP Number: **8270**

Written by: _____

Technical Review by: _____

Approved by: _____
Quality Assurance Manager

Approved by: _____
Laboratory Director

SOP Manual Control Number: _____

1.0 SCOPE/APPLICATION

1.1 Method SW-846 8270 is used in the determination of solvent-extractable semivolatile organic compounds in many types of solid waste matrices, soils, and groundwater. Most base-neutral and acidic organic compounds which are soluble in methylene chloride and capable of being eluted in a gas chromatograph without derivatization can be quantitated. Target compounds determined by this method are listed in Table 1.0

1.2 The following compounds require special treatment when being determined by this method.

1.2.1 Benzidine is subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.

1.2.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.2.3 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, N-Nitrosodimethylamine, 2-Naphthylamine, 4,6-Dinitro-2-Methylphenol, 4-Chloro-3-Methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, Pyridine, and Benzyl Alcohol are subject to erratic chromatographic behavior.

1.2.4 The analytes listed above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

1.2.5 N-nitrosodiphenylamine decomposes in the gas chromatograph and cannot be separated or differentiated from Diphenylamine. Both analytes are reported as a pair.

1.2.6 Azobenzene & 1,2-Diphenylhydrazine and 3 & 4-Methylphenol are reported as a pair.

1.3 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

Table 1.0
Target compounds with Detection Limits and Reporting Limits

PK#	***Compound Name***	***Water MDLs(ug/L)***			***Soil MDLs (mg/Kg)***	
		MDLs	***LOQs***		***MDLs***	***LOQs***
2	N-Nitrosodimethylamine	0.67	2.23		0.025	0.085
3	Pyridine	1.79	5.96		0.014	0.047
5	Aniline	0.11	0.37		0.009	0.029
6	Bis(2-chloroethyl) ether	0.08	0.27		0.012	0.041
8	Phenol	0.22	0.74		0.034	0.113
9	2-Chlorophenol	0.60	2.01		0.025	0.082
10	1,3-Dichlorobenzene	0.07	0.22		0.004	0.013
11	1,4-Dichlorobenzene	0.07	0.22		0.003	0.011
12	1,2-Dichlorobenzene	0.09	0.29		0.005	0.017
13	Benzyl alcohol	0.06	0.21		0.082	0.275
14	Bis(2-chloroisopropyl) ether	0.10	0.32		0.014	0.047
15	2-Methylphenol	0.26	0.88		0.032	0.107

PK#	***Compound Name***	***Water MDLs(ug/L)***			***Soil MDLs (mg/Kg)***	
		MDLs	***LOQs***		***MDLs***	***LOQs***
16	N-Nitrosopyrrolidine	0.07	0.22		0.013	0.043
17	Acetophenone	0.12	0.41		0.007	0.022
18	Hexachloroethane	0.17	0.56		0.008	0.025
19	N-Nitrosodi-n-propylamine	0.07	0.24		0.023	0.076
20	3 & 4-Methylphenol	0.47	1.56		0.051	0.171
23	Nitrobenzene	0.09	0.30		0.004	0.013
24	Isophorone	0.05	0.17		0.009	0.032
25	2-Nitrophenol	0.53	1.77		0.035	0.117
26	2,4-Dimethylphenol	0.43	1.44		0.021	0.068
27	Bis(2-chloroethoxy) methane	0.09	0.29		0.013	0.042
28	2,4-Dichlorophenol	0.57	1.89		0.026	0.088
29	1,2,4-Trichlorobenzene	0.07	0.24		0.004	0.012
30	Benzoic Acid	0.92	3.05		0.127	0.425
31	Naphthalene	0.08	0.26		0.003	0.010
32	4-Chloroaniline	0.09	0.30		0.003	0.008
33	2,6-Dichlorophenol	1.08	3.61		0.026	0.088
34	Hexachloropropene	0.13	0.44		0.19	0.62
35	Hexachlorobutadiene	0.11	0.38		0.009	0.029
36	4-Chloro-3-methylphenol	0.27	0.91		0.048	0.160
37	2-Methylnaphthalene	0.08	0.26		0.012	0.041
38	1-Methylnaphthalene	0.06	0.21		0.015	0.050
40	Hexachlorocyclopentadiene	0.08	0.28		0.016	0.053
41	1,2,4,5-Tetrachlorobenzene	0.07	0.25		0.007	0.024
42	2,4,6-Trichlorophenol	0.85	2.84		0.015	0.051
43	2,4,5-Trichlorophenol	0.72	2.40		0.058	0.193
45	2-Chloronaphthalene	0.07	0.22		0.004	0.014
46	2-Nitroaniline	0.56	1.88		0.006	0.021
47	Acenaphthylene	0.07	0.24		0.003	0.010
48	Dimethylphthate	0.08	0.27		0.013	0.043
49	2,6-Dinitrotoluene	0.08	0.28		0.009	0.030
50	Acenaphthene	0.09	0.29		0.004	0.012
51	3-Nitroaniline	0.49	1.63		0.009	0.030
52	2,4-Dinitrophenol	0.87	2.90		0.052	0.172
53	Dibenzofuran	0.07	0.24		0.016	0.054
54	2,4-Dinitrotoluene	0.07	0.23		0.021	0.071
55	4-Nitrophenol	0.52	1.72		0.093	0.309
56	2,3,4,6-Tetrachlorophenol	0.91	3.05		0.060	0.201
57	2-Naphthylamine	1.59	5.29		0.066	0.221
58	Fluorene	0.07	0.22		0.003	0.011
59	4-Chlorophenyl phenyl ether	0.08	0.27		0.004	0.013
60	Diethyl phthalate	0.09	0.29		0.016	0.052
61	4-Nitroaniline	0.09	0.30		0.006	0.019
64	4,6-Dinitro-2-methylphenol	0.64	2.14		0.045	0.151
65	N-Nitrosodiphenylamine & Diphenylamine	0.15	0.50		0.026	0.085
66	Azobenzene & 1,2-Diphenylhydrazine	0.12	0.39		0.009	0.031

PK#	*Compound Name*	*Water MDLs(ug/L)*			*Soil MDLs (mg/Kg)*	
		MDLs	*LOQs*		*MDLs*	*LOQs*
67	4-Bromophenyl phenyl ether	0.11	0.36		0.005	0.016
68	Hexachlorobenzene	0.06	0.21		0.017	0.058
69	Pentachlorophenol	0.91	3.02		0.023	0.078
70	Phenanthrene	0.08	0.25		0.015	0.051
71	Anthracene	0.06	0.21		0.003	0.011
72	Carbazole	0.07	0.22		0.003	0.011
73	Di-n-butyl phthalate	0.45	1.49		0.013	0.042
74	Fluoranthene	0.05	0.18		0.003	0.010
76	Benzidine	5.90	19.67		0.365	1.217
77	Pyrene	0.05	0.17		0.003	0.011
79	Butyl benzyl phthalate	0.12	0.40		0.017	0.057
80	3,3-Dichlorobenzidine	0.35	1.16		0.039	0.130
81	Benzo (a) anthracene	0.07	0.22		0.003	0.012
82	Chrysene	0.08	0.25		0.003	0.010
83	Bis (2-ethylhexyl) phthalate	0.23	0.77		0.020	0.067
84	Di-n-octyl phthalate	0.11	0.35		0.015	0.050
86	Benzo (b) fluoranthene	0.06	0.20		0.007	0.023
87	Benzo (k) fluoranthene	0.08	0.28		0.006	0.020
88	Benzo (a) pyrene	0.07	0.22		0.005	0.018
89	Indeno (1,2,3-cd) pyrene	0.09	0.30		0.005	0.017
90	Dibenz (a,h) anthracene	0.10	0.32		0.005	0.017
91	Benzo (g,h,i) perylene	0.10	0.33		0.007	0.024

Table 1.1
8270 Compound List

Codes (Tables 1):
S = Surrogates
I = Internal Standards
TM= Target Compounds
CCC= Calibration Check Compounds
SPCC= System Performance Check Compounds

Table 1.1						
PK#	*Compound Name*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion(s)*	*Code*
1	1,4-Dichlorobenzene d_4	4.96	1.00	152	115,78	I
2	N-Nitrosodimethylamine	2.66	0.54	74	42,43	TM
3	Pyridine	2.68	0.54	79	52	TM
4	2-Fluorophenol	3.56	0.72	112	64,57,92	S
5	Aniline	4.56	0.92	93	66,65	TM
6	Bis(2-chloroethyl) ether	4.62	0.93	93	95	TM
7	Phenol d_5	4.48	0.90	99	42,71,100	S

Table 1.1

PK#	*Compound Name*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion(s)*	*Code*
8	Phenol	4.49	0.91	94	65,66	CCC
9	2-Chlorophenol	4.69	0.95	128	130	TM
10	1,3-Dichlorobenzene	4.89	0.98	146	148,113	TM
11	1,4-Dichlorobenzene	4.98	1.00	146	148	CCC
12	1,2-Dichlorobenzene	5.17	1.04	146	148,113	TM
13	Benzyl alcohol	5.12	1.03	108	79,77,91	TM
14	Bis(2-chloroisopropyl) ether	5.31	1.07	45	121	TM
15	2-Methylphenol	5.26	1.06	108	107	TM
16	N-Nitrosopyrrolidine	5.44	1.10	100	41,42	TM
17	Acetophenone	5.47	1.10	105	77,51	TM
18	Hexachloroethane	5.58	1.13	117	201,199	TM
19	N-Nitrosodi-n-propylamine	5.47	1.10	70	42,130	SPCC
20	3 & 4-Methylphenol	5.46	1.10	108	107	TM
21	Naphthalene d_8	6.39	1.00	136	68,108,54	I
22	Nitrobenzene d_5	5.64	0.88	82	128,54,98	S
23	Nitrobenzene	5.66	0.89	77	123,65	TM
24	Isophorone	5.93	0.93	82	138	TM
25	2-Nitrophenol	6.01	0.94	139	109,65	CCC
26	2,4-Dimethylphenol	6.06	0.95	107	122,121	TM
27	Bis(2-chloroethoxy) methane	6.16	0.96	93	95,123,63	TM
28	2,4-Dichlorophenol	6.25	0.98	162	164,98	CCC
29	1,2,4-Trichlorobenzene	6.34	0.99	180	182,145	TM
30	Benzoic Acid	6.17	0.96	105	122,77,51	TM
31	Naphthalene	6.41	1.00	128	129,127	TM
32	4-Chloroaniline	6.47	1.01	127	129,65,92	TM
33	2,6-Dichlorophenol	6.47	1.01	162	164,98	TM
34	Hexachloropropene	6.49	1.02	213	211,215	TM
35	Hexachlorobutadiene	6.53	1.02	225	223,227	CCC
36	4-Chloro-3-methylphenol	6.89	1.08	107	144,142	CCC
37	2-Methylnaphthalene	7.01	1.10	141	142	TM
38	1-Methylnaphthalene	7.09	1.11	141	142	TM
39	Acenaphthene d_{10}	7.77	1.00	164	162,160	I
40	Hexachlorocyclopentadiene	7.14	0.92	237	235,272	SPCC
41	1,2,4,5-Tetrachlorobenzene	7.14	0.92	216	214,179	CCC
42	2,4,6-Trichlorophenol	7.23	0.93	196	198,200	TM
43	2,4,5-Trichlorophenol	7.26	0.93	196	198	TM
44	2-Fluorobiphenyl	7.30	0.94	172	171,170	S
45	2-Chloronaphthalene	7.38	0.95	162	127,164	TM
46	2-Nitroaniline	7.46	0.96	65	92,138,80	TM
47	Acenaphthylene	7.68	0.99	152	151,153	TM
48	Dimethylphthate	7.58	0.98	163	194,164	TM
49	2,6-Dinitrotoluene	7.63	0.98	165	89	TM
50	Acenaphthene	7.79	1.00	153	152	CCC
51	3-Nitroaniline	7.74	1.00	138	92	TM
52	2,4-Dinitrophenol	7.81	1.00	184	63,154	SPCC
53	Dibenzofuran	7.90	1.02	168	139	TM
54	2,4-Dinitrotoluene	7.89	1.02	165	89	TM

Table 1.1

PK#	*Compound Name*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion(s)*	*Code*
55	4-Nitrophenol	7.85	1.01	109	139,65	SPCC
56	2,3,4,6-Tetrachlorophenol	7.98	1.03	232	194,234,230	TM
57	2-Naphthylamine	8.01	1.03	143	115,116	TM
58	Fluorene	8.12	1.04	166	165,167	TM
59	4-Chlorophenyl phenyl ether	8.12	1.04	204	206,141	TM
60	Diethyl phthalate	8.04	1.04	149	177,150	TM
61	4-Nitroaniline	8.14	1.05	138	108,92,139	TM
62	2,4,6-Tribromophenol	8.29	1.06	330	332,141,222	S
63	Phenanthrene d_{10}	8.71	1.00	188	94,80,187	I
64	4,6-Dinitro-2-methylphenol	8.15	0.94	198	51,105	TM
65	N-Nitrosodiphenylamine & Diphenylamine	8.20	0.94	169	168,167	CCC
66	Azobenzene & 1,2-Diphenylhydrazine	8.22	0.94	182	152,77	TM
67	4-Bromophenyl phenyl ether	8.42	0.97	248	250,141	TM
68	Hexachlorobenzene	8.46	0.97	284	142,249	TM
69	Pentachlorophenol	8.58	0.99	266	264,268	CCC
70	Phenanthrene	8.72	1.00	178	179,176	TM
71	Anthracene	8.76	1.00	178	176,179	TM
72	Carbazole	8.85	1.02	167	166,139	TM
73	Di-n-butyl phthalate	9.02	1.04	149	150,104	TM
74	Fluoranthene	9.46	1.09	202	101	CCC
75	Chrysene d_{12}	10.41	1.00	240	120,236,106	I
76	Benzidine	9.52	0.92	184	183,185	TM
77	Pyrene	9.61	0.92	202	100,101	TM
78	Terphenyl d_{14}	9.68	0.93	244	122,212,245	S
79	Butyl benzyl phthalate	9.98	0.96	149	91,206	TM
80	3,3-Dichlorobenzidine	10.36	1.00	252	254	TM
81	Benzo (a) anthracene	10.40	1.00	228	229,226	TM
82	Chrysene	10.42	1.00	228	226,229	TM
83	Bis (2-ethylhexyl) phthalate	10.34	0.99	149	167,279	TM
84	Di-n-octyl phthalate	10.80	1.04	149	150	CCC
85	Perylene d_{12}	11.53	1.00	264	260,265,263,	I
86	Benzo (b) fluoranthene	11.20	0.97	252	253,125	TM
87	Benzo (k) fluoranthene	11.22	0.97	252	253,125	TM
88	Benzo (a) pyrene	11.49	1.00	252	253,125	CCC
89	Indeno (1,2,3-cd) pyrene	12.64	1.10	276	138	TM
90	Dibenz (a,h) anthracene	12.65	1.10	278	139,279	TM
91	Benzo (g,h,i) perylene	12.97	1.12	276	138,277	TM

Table 1.2
Extra Analyte List

PK#	*Compound*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion*	*Code*
92	N-Nitrosodiethylamine	3.48	0.75	102	57,56	TM

PK#	*Compound*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion*	*Code*
93	2-Chloro-5-Methylphenol	5.41	0.88	107	142,77	TM
94	2,5-Dichlorophenol	6.01	0.98	162	164,63,99	TM
95	2,3-Dichlorophenol	6.04	0.98	162	126,63,164,	TM
96	3&4-Chlorophenol	6.12	1.00	128	130,65,100	TM
97	N-Nitroso-di-n-butylamine	6.54	1.07	57	84,41,99	TM
98	2-Methyl-4-Chlorophenol	6.57	1.07	107	142,77	TM
99	3,4-Dichlorophenol	7.22	0.96	162	164,99,63	TM
100	2,5-Dinitrophenol	7.45	0.99	184	63,53,39	TM
101	Pentachlorobenzene	7.66	1.01	250	251,252,108	TM

Note: Retention time shifts can occur when instrument maintenance is performed.
Shifts in the retention times are reflected in the analytical method.

Table 1.3
Internal Standard For Each Target Compound

1,4-Dichlorobenzene d_4	**Naphthalene d_8**	**Acenaphthene d_{10}**	**Phenanthrene d_{10}**	**Chrysene d_{12}**	**Perylene d_{12}**
N-Nitrosodimethylamine	Nitrobenzene d_5	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Benzidine	Benzo (b) fluoranthene
Pyridine	Nitrobenzene	1,2,4,5-Tetrachlorobenzene	N-Nitrosodiphenylamine & Diphenylamine	Pyrene	Benzo (k) fluoranthene
2-Fluorophenol	Isophorone	2,4,6-Trichlorophenol	Azobenzene & 1,2-Diphenylhydrazine	Terphenyl d_{14}	Benzo (a) pyrene
Aniline	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenyl phenyl ether	Butyl benzyl phthalate	Indeno (1,2,3-cd) pyrene
Bis(2-chloroethyl) ether	2,4-Dimethylphenol	2-Fluorobiphenyl	Hexachlorobenzene	3,3-Dichlorobenzidine	Dibenz (a,h) anthracene
Phenol d_5	Bis(2-chloroethoxy) methane	2-Chloronaphthalene	Pentachlorophenol	Benzo (a) anthracene	Benzo (g,h,i) perylene
Phenol	2,4-Dichlorophenol	2-Nitroaniline	Phenanthrene	Chrysene	
2-Chlorophenol	1,2,4-Trichlorobenzene	Acenaphthylene	Anthracene	Bis (2-ethylhexyl) phthalate	
1,3-Dichlorobenzene	Benzoic Acid	Dimethylphthate	Carbazole	Di-n-octyl phthalate	
1,4-Dichlorobenzene	Naphthalene	2,6-Dinitrotoluene	Di-n-butyl phthalate		
1,2-Dichlorobenzene	4-Chloroaniline	Acenaphthene	Fluoranthene		
Benzyl alcohol	2,6-Dichlorophenol	3-Nitroaniline			
Bis(2-chloroisopropyl) ether	Hexachloropropene	2,4-Dinitrophenol			
2-Methylphenol	Hexachlorobutadiene	4-Nitrophenol			
N-Nitrosopyrrolidine	4-Chloro-3-methylphenol	2-Naphthylamine			
Acetophenone	2-Methylnaphthalene	Fluorene			
N-Nitrosodi-n-propylamine	1-Methylnaphthalene	4-Chlorophenyl phenyl ether			
3 & 4-Methylphenol		Diethyl phthalate			
Hexachloroethane		Dibenzofuran			
		2,4-Dinitrotoluene			
		4-Nitroaniline			
		2,4,6-Tribromophenol			
		2,3,4,6-			

1,4-Dichlorobenzene d_4	Naphthalene d_8	Acenaphthene d_{10}	Phenanthrene d_{10}	Chrysene d_{12}	Perylene d_{12}
		Tetrachlorophenol			

Table 1.4
Laboratory Control Sample Limits

	Inhouse					QSM			
	Water (%rec)		Soil (%rec)			Water (%rec)		Soil (%rec)	
Analyt	Lower Limit	Upper Limit	Lower Limit	Upper Limit		Lower Limit	Upper Limit	Lower Limit	Upper Limit
N-Nitrosodimethylamine	46	84	23	141		25	110	20	115
Pyridine	1	78	16	121					
Aniline	15	118	14	110					
Bis(2-chloroethyl) ether	56	105	45	126		35	110	40	105
Phenol	22	53	40	122		0	115	40	100
2-Chlorophenol	51	112	56	116		35	105	45	105
1,3-Dichlorobenzene	47	91	43	116		30	100	40	100
1,4-Dichlorobenzene	48	89	44	116		30	100	35	105
1,2-Dichlorobenzene	49	93	46	119		35	100	45	95
Benzyl alcohol	50	91	20	115		30	110	20	125
Bis(2-chloroisopropyl) ether	51	113	45	131		25	130	20	115
2-Methylphenol	52	93	47	128		40	110	40	105
N-Nitrosopyrrolidine	66	104	59	110					
Acetophenone	50	136	48	126					
Hexachloroethane	46	93	48	117		30	95	35	110
N-Nitrosodi-n-propylamine	61	110	54	124		35	130	40	115
3 & 4-Methylphenol	18	111	31	142		30	110	40	105
Nitrobenzene	64	101	51	116		45	110	40	115
Isophorone	63	99	50	107		50	110	45	110
2-Nitrophenol	52	119	58	110		40	115	40	110
2,4-Dimethylphenol	49	112	55	119		30	110	30	105
Bis(2-chloroethoxy) methane	64	104	50	122		45	105	45	110
2,4-Dichlorophenol	53	120	47	126		50	105	45	110
1,2,4-Trichlorobenzene	53	97	45	122		35	105	45	110
Benzoic Acid	1	125	27	57		0	125	0	110
Naphthalene	58	105	47	129		40	100	40	105
4-Chloroaniline	69	110	13	116		15	110	10	95
2,6-Dichlorophenol	68	112	48	126					
Hexachloropropene	40	119	45	136					
Hexachlorobutadiene	42	97	45	120		25	105	40	115
4-Chloro-3-methylphenol	69	111	60	115		45	110	45	115
2-Methylnaphthalene	47	96	42	112		45	105	45	105
1-Methylnaphthalene	70	130	70	130					
Hexachlorocyclopentadiene	36	106	30	137					
1,2,4,5-Tetrachlorobenzene	58	109	44	134					
2,4,6-Trichlorophenol	56	110	37	123		50	115	45	110
2,4,5-Trichlorophenol	44	125	60	115		50	110	50	110
2-Chloronaphthalene	59	100	48	119		50	105	45	105

	Inhouse					QSM			
	Water (%rec)		Soil (%rec)			Water (%rec)		Soil (%rec)	
Analyt	Lower Limit	Upper Limit	Lower Limit	Upper Limit		Lower Limit	Upper Limit	Lower Limit	Upper Limit
2-Nitroaniline	54	113	52	115		50	115	45	120
Acenaphthylene	58	109	45	128		50	105	45	105
Dimethylphthate	67	102	52	113		25	125	50	110
2,6-Dinitrotoluene	74	129	58	124		50	115	50	110
Acenaphthene	64	105	50	123		45	110	45	110
3-Nitroaniline	59	119	39	111		20	125	25	110
2,4-Dinitrophenol	1	138	11	114		15	140	15	130
Dibenzofuran	60	96	47	113		55	105	50	105
2,4-Dinitrotoluene	62	117	57	112		50	120	50	115
4-Nitrophenol	11	65	19	124		0	125	15	140
2,3,4,6-Tetrachlorophenol	70	130	70	130					
2-Naphthylamine	37	187	26	140					
Fluorene	69	107	52	126		50	110	40	115
4-Chlorophenyl phenyl ether	68	105	51	123		50	110	45	110
Diethyl phthalate	73	118	60	124		40	120	50	115
4-Nitroaniline	54	121	38	112		35	120	35	115
4,6-Dinitro-2-methylphenol	37	124	14	139		40	130	30	135
N-Nitrosodiphenylamine & Diphenylamine	77	109	53	125		50	110	50	115
Azobenzene & 1,2-Diphenylhydrazine	72	103	49	119		55	115		
4-Bromophenyl phenyl ether	70	105	54	121		50	115	45	115
Hexachlorobenzene	70	111	54	122		50	110	45	120
Pentachlorophenol	39	117	19	140		40	115	25	120
Phenanthrene	75	107	53	126		50	115	50	110
Anthracene	78	110	57	128		55	110	55	105
Carbazole	73	119	54	123		50	115	45	115
Di-n-butyl phthalate	76	126	62	125		55	115	55	110
Fluoranthene	71	111	53	120		55	115	55	115
Benzidine	1	165	1	161					
Pyrene	68	110	50	123		50	130	45	125
Butyl benzyl phthalate	77	110	59	117		45	115	50	125
3,3-Dichlorobenzidine	74	126	35	114		20	110	10	130
Benzo (a) anthracene	72	112	60	119		55	110	50	110
Chrysene	71	111	57	119		55	110	55	110
Bis (2-ethylhexyl) phthalate	73	122	61	123		40	125	45	125
Di-n-octyl phthalate	70	117	57	124		35	135	40	130
Benzo (b) fluoranthene	72	121	57	120		45	120	45	115
Benzo (k) fluoranthene	65	121	50	136		45	125	45	125
Benzo (a) pyrene	73	119	58	123		55	110	50	110
Indeno (1,2,3-cd) pyrene	59	130	46	125		45	125	25	135
Dibenz (a,h) anthracene	54	129	48	125		40	125	40	125
Benzo (g,h,i) perylene	55	136	42	133		40	125	40	125

Table 1.5
Matrix Spike/Matrix Spike Duplicate Control Limits

	Inhouse						QSM					
	Water (*rec)			Soil (%rec)			Water (%rec)			Soil (%rec)		
Analyt	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**
N-Nitrosodimethylamine	39	121	28	18	138	39	25	110	30	20	115	30
Pyridine	1	115	54	1	114	52						
Aniline	1	132	48	1	81	60						
Bis(2-chloroethyl) ether	48	116	29	48	105	31	35	110	30	40	105	30
Phenol	1	126	41	53	102	35	0	115	30	40	100	30
2-Chlorophenol	56	110	25	51	108	35	35	105	30	45	105	30
1,3-Dichlorobenzene	29	114	28	31	110	38	30	100	30	40	100	30
1,4-Dichlorobenzene	37	103	28	32	108	36	30	100	30	35	105	30
1,2-Dichlorobenzene	28	116	27	38	108	36	35	100	30	45	95	30
Benzyl alcohol	31	134	28				30	110	30	20	125	30
Bis(2-chloroisopropyl) ether	46	129	27	41	115	41	25	130	30	20	115	30
2-Methylphenol	30	127	35	23	134	38	40	110	30	40	105	30
N-Nitrosopyrrolidine	51	119	26	52	111	32						
Acetophenone	38	141	27	43	127	33						
Hexachloroethane	27	121	33	13	134	32	30	95	30	35	110	30
N-Nitrosodi-n-propylamine	32	145	25	58	107	32	35	130	30	40	115	30
3 & 4-Methylphenol	1	158	38	19	150	34	30	110	30	40	105	30
Nitrobenzene	40	127	31	45	110	39	45	110	30	40	115	30
Isophorone	53	109	28	52	94	31	50	110	30	45	110	30
2-Nitrophenol	52	116	30	43	110	34	40	115	30	40	110	30
2,4-Dimethylphenol	3	116	30	35	119	47	30	110	30	30	105	30
Bis(2-chloroethoxy) methane	53	119	27	57	100	32	45	105	30	45	110	30
2,4-Dichlorophenol	54	112	32	51	113	32	50	105	30	45	110	30
1,2,4-Trichlorobenzene	44	106	31	47	107	36	35	105	30	45	110	30
Benzoic Acid	0	125	24	1	90	20	0	125	30	0	110	30
Naphthalene	40	122	29	56	107	33	40	100	30	40	105	30
4-Chloroaniline	21	134	31	1	94	57	15	110	30	10	95	30
2,6-Dichlorophenol	36	125	46	40	131	39						
Hexachloropropene	18	135	30	1	158	47						
Hexachlorobutadiene	34	109	38	37	112	32	25	105	30	40	115	30
4-Chloro-3-methylphenol	19	139	34	57	115	37	45	110	30	45	115	30
2-Methylnaphthalene	36	107	25	40	112	36	45	105	30	45	105	30
1-Methylnaphthalene	70	130	30	70	130	30						
Hexachlorocyclopentadiene	10	140	34	1	141	38						
1,2,4,5-Tetrachlorobenzene	46	123	25	51	117	33						
2,4,6-Trichlorophenol	20	135	46	19	121	32	50	115	30	45	110	30
2,4,5-Trichlorophenol	37	139	40	40	134	30	50	110	30	50	110	30
2-Chloronaphthalene	46	114	25	56	101	31	50	105	30	45	105	30

	Inhouse						QSM					
	Water (*rec)			Soil (%rec)			Water (%rec)			Soil (%rec)		
Analyt	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**	**LL**	**UL**	**RPD**
2-Nitroaniline	40	149	21	33	126	36	50	115	30	45	120	30
Acenaphthylene	50	121	24	54	109	30	50	105	30	45	105	30
Dimethylphthate	41	132	20	57	100	30	25	125	30	50	110	30
2,6-Dinitrotoluene	61	133	21	46	126	31	50	115	30	50	110	30
Acenaphthene	49	126	25	59	109	31	45	110	30	45	110	30
3-Nitroaniline	36	132	32	1	122	36	20	125	30	25	110	30
2,4-Dinitrophenol	1	148	50	0	118	36	15	140	30	15	130	30
Dibenzofuran	49	114	23	56	98	29	55	105	30	50	105	30
2,4-Dinitrotoluene	51	129	21	44	123	35	50	120	30	50	115	30
4-Nitrophenol	1	141	49	1	141	44	0	125	30	15	140	30
2,3,4,6-Tetrachlorophenol	70	130	30	70	130	30						
2-Naphthylamine	4	176	52	1	166	20						
Fluorene	50	130	20	59	115	31	50	110	30	40	115	30
4-Chlorophenyl phenyl ether	57	121	20	61	107	29	50	110	30	45	110	30
Diethyl phthalate	58	135	18	59	119	30	40	120	30	50	115	30
4-Nitroaniline	26	142	21	1	139	33	35	120	30	35	115	30
4,6-Dinitro-2-methylphenol	1	164	41	14	118	41	40	130	30	30	135	30
N-Nitrosodiphenylamine & Diphenylamine	34	135	25	29	129	34	50	110	30	50	115	30
Azobenzene & 1,2-Diphenylhydrazine	55	113	17	62	104	32	55	115	30			30
4-Bromophenyl phenyl ether	59	118	18	62	107	30	50	115	30	45	115	30
Hexachlorobenzene	54	119	18	59	111	30	50	110	30	45	120	30
Pentachlorophenol	1	147	33	23	126	27	40	115	30	25	120	30
Phenanthrene	50	130	13	54	119	29	50	115	30	50	110	30
Anthracene	57	131	14	54	122	29	55	110	30	55	105	30
Carbazole	51	128	19	42	125	34	50	115	30	45	115	30
Di-n-butyl phthalate	61	137	23	55	130	28	55	115	30	55	110	30
Fluoranthene	49	126	15	57	112	31	55	115	30	55	115	30
Benzidine	1	163	50	6	12	20						
Pyrene	52	133	15	45	123	29	50	130	30	45	125	30
Butyl benzyl phthalate	48	142	17	51	134	27	45	115	30	50	125	30
3,3-Dichlorobenzidine	3	148	29	1	117	53	20	110	30	10	130	30
Benzo (a) anthracene	50	130	16	43	137	36	55	110	30	50	110	30
Chrysene	34	138	16	31	135	36	55	110	30	55	110	30
Bis (2-ethylhexyl) phthalate	32	156	36	55	126	30	40	125	30	45	125	30
Di-n-octyl phthalate	18	159	27	54	129	30	35	135	30	40	130	30
Benzo (b) fluoranthene	40	135	23	51	139	43	45	120	30	45	115	30
Benzo (k) fluoranthene	44	140	23	43	140	38	45	125	30	45	125	30
Benzo (a) pyrene	42	138	19	44	138	33	55	110	30	50	110	30
Indeno (1,2,3-cd) pyrene	16	143	24	8	130	37	45	125	30	25	135	30
Dibenz (a,h) anthracene	15	148	22	6	133	36	40	125	30	40	125	30
Benzo (g,h,i) perylene	20	148	24	12	127	41	40	125	30	40	125	30

Note: Information on recovery limits and RPDs are generated through StarLIMS. The information tables are stored in H:\Quality Systems\QC\charting.

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous and soil matrices (reference methods SW846-3510 and 3545), concentration techniques that are suitable for preparing the extract, and the quantitative/qualitative analysis for the determination of target analytes by method SW846-8270.

2.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method applies for aqueous samples extracted by liquid-liquid separatory funnel (SW846-3510). Method applies for soil/sediment, and solid waste samples extracted by standard solvent extraction methods utilizing pressurized extraction techniques as heated pressurized fluid extraction (SW846-3545). This method includes the extraction for waste dilution samples.

2.3 The resultant extract is chemically dried and concentrated in a Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

2.4 Extracts for 8270 analysis can be subjected to a variety of cleanup steps, depending on the nature of the matrix interferences and target analytes. Suggested cleanup steps include Gel-Permeation Chromatography (GPC) cleanup (SW846 3640A), Acid-Base Partition (SW846-3650), Florisil (SW846-3620), and/or Silica Gel (SW846-3630) cleanup. After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with a mass spectrometer detector.

2.5 Identification of target analytes is accomplished by comparing their mass spectra with the spectra of certified commercially-prepared stock standards. Quantitation is accomplished by comparing the response of a major quantitation ion relative to an internal standard using a minimum of a five point calibration curve.

2.6 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

3.0 DEFINITIONS

3.1 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.2 Laboratory Control Spike (LCS): Milli-Q water (for water) or Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike is used to document the ability of

an analyst to generate acceptable precision and bias, to verify the analytical system performance, and to document method accuracy for each matrix.

3.3 Laboratory Control Spike Duplicate (LCSD): A duplicate Milli-Q water (for water) or Organic-Free Soil (for soil) is spiked with the target analytes and carried through the complete sample preparation and analytical procedure. The control spike duplicate is used to document the precision and bias of a method. This QC type is only performed when sufficient amount of sample is unavailable for performing a matrix spike and matrix spike duplicate.

3.4 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the precision and bias of a method in a given sample matrix.

3.5 Matrix Spike Duplicate (MSD): Intra-laboratory split samples spiked with identical concentration of target analytes. The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

3.6 Method Reporting Limit (MRL): The method reporting limit is a threshold value below which the laboratory reports a result as a non-detect. The highest value reported for the method reporting limit is dependant upon project-specific action or decision levels. Method reporting limits are adjusted based on the sample matrix and any sample dilution/concentrations when necessary.

3.7 Method Detection Limit (MDL): The method detection limit is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. Depending on project, specific MDLs are either in the form of an MDL check standard or the MDL is subject to extraction procedures. An acceptable MDL standard check must produce signals for the qualifier ions. An MDL standard injection must be made after each major instrument repair or changing of instruments (prior to sample analysis) to determine the performance sensitivity of the analysis.

3.8 Surrogate (SURR): Organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Its use is to monitor the performance of the extraction, cleanup (as needed), the analytical system, and the effectiveness of the method. The acid surrogates are at twice the concentration of the Base/Neutral surrogates. The acid surrogate compounds are: 2-Fluorophenol, Phenol d_5, and 2,4,6-Tribromophenol. The base/neutral surrogates are: Nitrobenzene d_5, 2-Fluorobiphenyl, and Terphenyl d_{14}.

3.9 Initial Calibration (ICAL): An analytical instrument is said to be calibrated when an instrumental response can be related to the concentration of an analyte. This relationship is depicted graphically and referred to as a “calibration curve”. Initial calibration curves must be established based upon the requisite number of standards identified within the method for each target analyte.

3.10 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis

of all target analytes each time the initial calibration is performed.

3.11 Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. A CCV must be made during each 12 hour shift.

3.12 DFTPP: Decafluorotriphenylphosphine. This compound is used in tuning the GC/MS. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Benzidine and Pentachlorophenol must have tailing factors less than 2. Breakdown of DDT to DDD and DDE must be less than 20%.

3.13 Internal standard (ISTD): Internal Standard involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample extract prior to injection. The ratio of the peak area of the target compound in the sample extract to the peak area of the internal standard in the sample extract is compared to a similar ratio derived for each calibration standard. The internal standards are; 1,4-Dichlorobenzene $d_{4,}$ Naphthalene $d_{8,}$ Acenaphthene $d_{10,}$ Phenanthrene $d_{10,}$ Chrysene d_{12} and Perylene $d_{12.}$

3.14 System Performance Check Compounds (SPCC): SPCCs are system performance compounds that are a part of the Continuing Calibration Verification standard (CCV). The SPCCs must meet a minimum response factor of 0.050. The SPCC criteria also apply to the average response factor of the initial calibration curve.

3.15 Calibration Check Compounds (CCC): CCCs are calibration check compounds that are a part of the Continuing Calibration Verification standard (CCV). The CCC percent difference must be less than or equal to 20%.

3.16 Instrument Blanks (IB): Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it can be followed by the analysis of an instrument blank (methylene chloride + Internal Standards) to check for cross-contamination.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and/or purification of solvents by distillation in all-glass systems will be necessary. Refer to each method for specific guidance on quality control procedures.

4.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, will cause degradation of certain analytes.

4.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample or dilution of the sample will be necessary.

5.0 SAFETY

5.1 Protective clothing: safety glasses, gloves, apron and/or lab coat, long pants, and protective shoes, shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

5.2 The toxicity of chemicals used in this method has not been precisely defined. Each chemical shall be treated as a potential health hazard, and exposure to these chemicals shall be minimized.

6.0 APPARATUS & MATERIALS

Apparatus

6.1 GC-MS system - Hewlett Packard 6890 GC/7683 autosampler/5973 MSD - An analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including syringes, analytical column, mass spectrometer detector, auto sampler, electronic pressure control, vacuum pumps, and HP Chemstation data acquisition system.

6.1.1 Carrier Gas: He at 1.2 mL/min (constant flow)
Injector Temperature: 250º C
Mode: Pulsed Splitless
Inj. Volume: 0.5 uL
Pressure: 8.9 psi
Pulse Pressure: 30.0 psi
Pulse Time: 0.4 min
Purge Flow: 50.0 ml/min
Purge Time: 0.38 min
Total Flow: 53.5 ml/min
Gas Saver On: 20ml/min at 2 min
Oven: Initial – 35º (hold for 0.45 min)
Ramp – 30º/min
Final –70º (hold for 0 min)
Ramp – 14º/min
Final – 120º (hold for 0 min)
Ramp – 45º/min
Final – 220º (hold for 0 min)
Ramp – 40º/min
Final – 280º (hold for 0 min)
Ramp – 30º/min

Final – 325º (hold for 3.6 min)

6.1.2 MS Interface: 280º
MS Source: 230º
Mass range: 35-500 amu
Scan time: 0.317 sec/scan

6.2 30m x 0.25 mm ID, 0.25 um. (J&W DB-5.625 or equivalent).

6.3 Water bath – heated and capable of accepting a Kuderna-Danish apparatus.

6.4 Dionex ASE 200 Accelerated Solvent Extractor.

6.5 Organomation Nitrogen blowdown concentrator.

6.6 Analytical balance capable of accurately weighing to the nearest 0.01 gram (for sample preparation).

6.7 Oven.

Glassware

6.8 Separatory funnel - 2000 mL glass with Teflon stopcock

6.9 Kuderna-Danish (K-D) apparatus:
6.9.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).
6.9.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).
6.9.3 Snyder column- Three-ball macro (Fisher # K503000-0121).
6.9.4 Springs or Teflon clamps to attach concentrator tube to evaporation.

6.10 Graduated cylinder (Class A) - 1000 mL. (Fisher 08-559G).

6.11 Beaker - 250 mL and 600 mL.

6.12 Vials - 2.0 mL and 60 mL amber screw cap vials with Teflon lined caps (Fisher # 03-377-10B).

6.13 Pasteur Pipets (Corning # 7095B-5X).

6.14 Funnels – glass

6.15 Volumetric flask – (Class A) 2, 5, 10, 25, 50, and 100 mL.

6.16 Syringes 10 uL, 25 uL, 100 uL, 500 uL, and 1,000 uL.

Reagents

6.17 Deionized water (Milli-Q processed).

6.18 Sodium sulfate (granular, anhydrous 60/120 mesh, Fisher # S415-212). Purify by heating to 400°C in a shallow glass tray for 4 hours.

6.19 Silica sand- hydrocarbon free. Purify by heating to 400°C in a shallow glass tray for 4 hours.

6.20 Methylene chloride, pesticide grade.

6.21 Acetone, pesticide grade.

6.22 Methanol, pesticide grade.

6.23 Sulfuric Acid (Certified ACS)/Deionzied Water-1:1(v/v).

6.24 Sodium Hydroxide- 10 N (Certified ACS).

6.25 Diatomaceous earth, pelletized (Dionex # 062819).

6.26 Nitrogen (4.8 rating).

6.27 Helium (4.7 rating).

NOTE: Chemicals are stored at room temperature and in solvent cabinets. The solvents are used in well-ventilated areas. The organic solvents have a shelf life of one year from the date of receipt.

Materials

6.28 Boiling chips, carborundum, approximately 10/40 mesh (methylene chloride rinsed) (G. Frederick Smith Chemical Company, Catalog #198).

6.29 pH indicator paper- pH 0-14 (Whatman Catalog #2613991).

6.30 Dionex ASE Filters (Catalog #049458).

6.31 Filter- Glass Microfibre 12.5 cm GF/A (Whatman # 1827-125).

6.32 Aluminum foil.

6.33 Spatulas- stainless steel.

7.0 STANDARDS AND SPIKES

7.1 Preparation of standards is documented in the GC/MS standards logbook. Each standard is labeled by prep date to allow for tracking. Opened stock standards expire in six months or sooner if comparison with quality control check samples indicate a problem. Leftover stock standards are saved in a capped vial in the original box in the freezer. Any subsequent dilutions made from the opened vial expire six months from the original opening. The cracking date of the stock standard vial will be recorded on the label along with the six month expiration date.

7.2 Stock Standards – Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at –10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Opened stock standards expire in six months or sooner if comparison with quality control check samples

indicate a problem (Not to exceed the manufactures expiration date).The following list of stock standards are commercially prepared standards, which are certified by the manufacturer, such as;

BIG BN-2: Protocol Part # BIG BN-2 at 2000 ug/ml
HICAL-ACIDS: Protocol Part # HICAL-ACIDS at 2000 ug/ml
Benzidines: Protocol Part # 605X at 2000 ug/ml
Balance Mix A: Protocol Part # SV-X at 2000 ug/ml
Custom Balance Mix B: Protocol Part # XCT-CM-2 at 2000 ug/ml
8270 Surrogate Mix BN: Restek #31062 5000 ug/ml
8270 Surrogate Mix AE: Restek #31063 10000 ug/ml
DFTPP: RESTEK # 31615 at 1000 ug/ml

7.3 Intermediate Stock Standards: These standards are diluted stock standards so that the concentration levels are manageable for the preparation of working standards. The 8270 intermediate standard is prepared at an optimum level for the preparation of the working stock standard. Each 8270 target compound (or Surrogate) is at a concentration of 100.0 ug/ml. in methylene chloride with the following exceptions: compounds that co-elute (listed in sec 1.2.5 and 1.2.6) are at a concentration of 200.0 ug/mL. For example, Azobenzene and 1,2-Diphenylhydrazine are each in the stock solution at 100.0 ug/mL. They are reported as a pair (Azobenzene&1,2-Diphenylhydrazine) with a concentration of 200.0 ug/mL.

Table 2.0
Intermediate Stock Standard

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
BIG BN	2000	0.500	10.0	100.0
HICAL-ACIDS	2000	0.500	10.0	100.0
Benzidines	2000	0.500	10.0	100.0
Balance Mix A	2000	0.500	10.0	100.0
Balance Mix B	2000	0.500	10.0	100.0
8270 Surr Mix BN	5000	0.200	10.0	100.0
8270 Surr Mix AE	10000	0.100	10.0	100.0

7.4 Calibration standards: An initial calibration of the listed analytes in Table 1.0 is performed using a minimum of 5 points. The following concentrations correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. Standards are made by taking aliquots of the intermediate standard and diluting to volume in methylene chloride. The following levels are repeated across all 8270 compounds. Note: due to low instrument response the following compounds are not calibrated from level 1: Benzoic acid, 2,4-Dinitrophenol, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol and Pentachlorophenol.

Level 1 1.00 ug/ml
Level 2 5.00 ug/ml
Level 3 10.0 ug/ml
Level 4 20.0 ug/ml

Level 5 30.0 ug/ml
Level 6 40.0 ug/ml
Level 7 50.0 ug/ml

Table 2.1
Initial Calibration Points for the 8270 Linearity

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
1	100.0	0.100	10	1.0
2	100.0	0.500	10	5.0
3	100.0	1.000	10	10.0
4	100.0	2.000	10	20.0
5	100.0	1.500	5	30.0
6	100.0	2.000	5	40.0
7	100.0	2.500	5	50.0

7.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target compounds at 20.0 ug/ml (40.0 ug/mL for Azobenzene&1,2-Diphenylhydrazine, N-nitrosodiphenylamine&Diphenylamine) each time the initial calibration is performed. Standards are made by taking aliquots of the ICV intermediate standard and diluting to volume in methylene chloride. The ICV stock standard is prepared in the same manner as the primary intermediate stock standard. The ICV working standard will be prepared at the time of initial calibration and have a shelf life of one week.

Table 2.2
ICV Working 8270 Standard

Working ICV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
ICV1	100.0	1.000	5.0	20.0

7.6 Calibration Verification Standard (CCV): A working standard solution for 8270 at a concentration of 20.0 ug/ml and is used to check the validity of a calibration curve on a daily basis. Standard is made by taking an aliquot of the intermediate standard and diluting it to volume in methylene chloride. The CCV is prepared weekly and stored at 4ºC.

Table 2.3
CCV Working 8270 Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270	100.0	1.00	5.0	20.0

7.7 Surrogate standard: Commercially prepared certified solutions of 2-Fluorophenol, Phenol d_5, and 2,4,6-Tribromophenol at 10000 ug/ml (AE Surrogates) and Nitrobenzene d_5, 2-Fluorobiphenyl, and Terphenyl d_{14} (BN Surrogates) are diluted in methanol to produce a working surrogate solution of 40/20 ug/mL (AE/BN). 1.0 mL is added to each sample. The surrogate concentration is normalized to 100% from the spiking solution in the initial calibration. This will provide percent recoveries that transfer directly to LIMS.

Table 2.4
Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 Surr. Mix BN	5000.0	0.400	100.0	20.0
8270 Surr. Mix AE	10000.0	0.400	100.0	40.0

7.8 Spiking standards (matrix and control samples): Prepare a spiking solution in methanol that contains target compounds for water and sediment / soil samples. 1.0 ml is added to quality control and matrix spike samples. The concentration of these compounds are five times higher for waste samples. If other compounds of interest are to be monitored they can be added at an appropriate level and noted in the standard preparation log. If client requests, spiking solution can be altered to match the target analytes of interest.

Table 2.5
Compound Spiking Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270	2000	0.500	50.0	20.0

7.9 DFTPP: Decafluorotriphenylphosphine. This compound is used in tuning the GC/MS. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness.

Table 2.6
DFTPP Standard Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
DFTPP	1000	0.250	10.0	25.0

NOTE: All standards are stored at -10ºC. Opened stock standards expire in six months or sooner if comparison with quality control check samples indicate a problem. Intermediate standards expire six months from the prep date. An intermediate stock standard or working standard shall not exceed expiration date criteria. All subsequent standards made from the intermediate stock standards expire on the same date as the working stock standard. If more than one standard is added to a solution the expiration date will be the same as the stock standard with the earliest expiration date

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. The soil samples must be extracted within 14 days and water samples must be extracted in 7 days, from the date of collection.

8.2 Sample extracts are to be stored under refrigeration in the dark and analyzed within 40 days of extraction.

9.0 QUALITY CONTROL

9.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

9.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in µg/L and the relative standard deviation (RSD) of the recovery 8270 target compounds. In addition to each set of IDCs, a blind laboratory spike will be performed. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000, sec. 8.4.9).

9.3 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SURR, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Client and/or Project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 4) also lists recovery limits specific to the method/project/program.

10.0 CALIBRATION

10.1 Calibration

10.1.1 The initial calibration for SW-846 chromatographic methods involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument.

10.1.2 For each compound and surrogate of interest, prepared calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methylene chloride.

10.1.3 The lowest concentration calibration standard, that is analyzed during an initial calibration curve, establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

10.1.4 Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the response of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area or height of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and is also known as a relative response factor in other methods.

10.1.4.1 Internal standards are recommended in SW846-8270. These internal standards are: 1,4-Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12}. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

10.1.4.2 In preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. 10uL of a solution containing the internal standards at a concentration of 2,000 ug/mL is added to each 1 mL of standard or sample extract. This results in an internal standard concentration of 20.0 ug/mL on the instrument. The mass of each internal standard added to each sample extract immediately prior to injection into the instrument must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts is such that minimal dilution of the extract occurs (e.g., 10 ul of solution added to a 1 ml final extract results in only a negligible 0.1% change in the final extract volume which can be ignored in the calculations).

10.1.4.3 An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard shall produce an instrument response (area counts) that is no more than 100 times that produced by the lowest concentration

of the least responsive target analyte associated with the internal standard. This results in a minimum response factor of approximately 0.01 for the least responsive target compound.

10.1.5 For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows;

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Concentration of the analyte or surrogate in ug/mL.

C_{is} = Concentration of the internal standard in ug/mL.

10.1.6 Linear calibration using the average response factor. Response factors are a measure of the slope of the calibration relationship and assume that the curve passes through the origin. Under ideal conditions, the factors will not vary with the concentration of the standard that is injected into the instrument. In practice, some variation is to be expected. However, when the variation, measured as the relative standard deviation (RSD), is less than or equal to 15%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. To evaluate the linearity of the initial calibration, calculate the RF, the standard deviation, and the relative standard deviation.

$$\text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (RF_I + \overline{RF})^2}{n-1}\right)}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

10.1.7 The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C sec. 11.5).

When ARFs are not acceptable, results are sometimes calculated using linear (1st order) regression curves and/or quadratic (2nd order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5).

10.1.8 Linear Calibration: If the RSD of the calibration factor is greater than 15% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

10.1.9 The use of origin (0,0) as a calibration point is not allowed. However, most data systems and many commercial software packages will allow the analyst to "force" the regression through zero. This is not the same as including the origin as a fictitious point in the calibration. It can be appropriate to force the regression through zero for some calibrations (SW-846 Method 8000C sec. 11.5.2.1). The use of linear regression cannot be used as a rationale for reporting results below the calibration range.

10.1.10 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it can be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

10.1.11 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Linear regression employs five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards. The coefficient of determination (COD) is calculated as follows:

$$COD = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

y_{obs} = Observed response (area) for each concentration of the calibration curve.

$\bar{y}$ = Mean observed response from the initial calibration.
Y_I = Calculated response at each concentration from the initial calibrations.
n = Total number of calibration points (6 points for quadratic equation).
p = Number of adjustable parameters in the polynomial.

10.1.12 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0 In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99. Weighting in a calibration model can significantly improve the ability of the least squares regression to fit the data calibrations (SW-846 Method 8000C sec. 11.5.3).

10.2 Calibration Criteria

10.2.1 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so a 25 ng injection of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 3. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

Table 3.0
DFTPP Mass Spectrum

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
69	<100% of mass 198
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1.0% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

10.2.2 To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background

noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP.

10.2.3 The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and Pentachlorophenol shall be present at their normal responses and peak tailing shall be evaluated. Benzidine and Pentachlorophenol must have tailing factors less than 2.

10.2.4 Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels and are prepared from the working standard dilutions of intermediate stock standards. One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels shall correspond to the expected range of concentrations found in real samples and shall contain each analyte for detection by this method. If the measured relative standard deviation (RSD) is less than or equal to 15%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. Linear Calibration: If the RSD of the calibration factor is greater than 15% over the calibration range, then linearity though the origin cannot be assumed. In this case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation. In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range.

10.2.5 System Performance Check Compounds (SPCC): Are system performance compounds that are a part of the Continuing Calibration Verification standard (CCV). A CCV must be made during each 12 hour shift. The SPCCs must meet a minimum response factor of 0.050. The SPCCs criteria also apply to the average response factor of the initial calibration curve.

10.2.6 Calibration Check Compounds (CCC): Are calibration check compounds that are a part of the Continuing Calibration Verification standard (CCV). In the initial calibration curve, the percent RSD of the CCCs must be less than or equal to 30%. A CCV must be analyzed during each 12 hour shift. The CCCs percent difference must be less than or equal to 20%.

10.2.7 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed. The SPCCs must meet minimum response factor of 0.050. The percent difference of the CCCs must be less than or equal to 20%.

10.2.8 Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during

sample analysis. The SPCCs must meet minimum response factor of 0.050. The percent difference of the CCCs must be less than or equal to 20%.

10.2.9 The relative retention time (RRT) of each compound in each calibration standard shall agree within 0.06 RRT units.

11.0 PREPARATION PROCEDURES

11.1 Water Extraction (Method SW-846,3510)

11.1.1 Pre-rinse all glassware to be used in the extraction with methylene chloride (Pesticide Grade).

11.1.2 Mark the meniscus on the bottle for later determination of sample volume (see sec. 11.1.11). From the glass sample collection bottle, quantitatively transfer sample into a 2 liter separatory funnel.

11.1.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding one liter of Milli-Q water to a 2 liter separatory funnel.

11.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample from the glass sample collection bottle, quantitatively transfer half of the sample into a 2 liter separatory funnel and label MS, and transfer the other half of the sample into another 2 liter separatory funnel and label MSD. If there is no sample to perform a matrix spike/matrix spike duplicate a laboratory control spike duplicate must be performed.

11.1.5 Check and adjust the pH to <2 by with 1:1 sulfuric acid.

11.1.6 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 1.0 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.1.7 Add 60 mLs of methylene chloride to the sample's separatory funnel. Extract the sample shaking vigorously for two minutes, venting frequently.

11.1.8 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Decant the lower layer into a 500 ml beaker. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and can include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Check sample pH to insure acidic conditions.

11.1.9 Repeat the extraction two more times using fresh 60 mL portions of methylene chloride.

11.1.10 After the third extraction of the acidified sample, adjust the pH >12 with 10N

sodium hydroxide.

11.1.11 Determine the sample volume by filling the sample bottle to the mark (11.1.2) with water and transferring it to a "Class A" 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet.

11.1.12 Refer to section 11.4 for sample concentration.

11.2 **Soil Extraction (Method SW-846, 3545)**

11.2.1 Preparing the extraction cell for use: Pre-rinse extraction cell (tube and caps) with methylene chloride. Attach the screw fit tube cap of the soil extractor to the end of the tube without the serial number engraved on the side. Using a filter rod, push one 19.8mm filter through the open end of the tube until it resides flush on the bottom of the screwed end.

11.2.2 Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1mm sieve or can be extruded through a 1mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make sample more amenable to grinding.

11.2.4 Gummy, fibrous, or oily materials not amenable to grinding shall be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make sample easier to mix.

11.2.5 (Refer to SOP FO-10 for subsampling guidance). Weigh approximately 10 g of sample to the nearest 0.1 g into a 250-mL beaker and record the final weight. Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples shall be a free flowing powder. If sample is not free flowing add more diatomaceous earth until the sample has a dry texture. This powder is so mixed that it will allow the sample to pass through a 2 mm sieve.

11.2.6 Transfer the ground sample to an extraction cell of appropriate size for the aliquot.

11.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10g of sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

11.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh three individual 10 grams aliquots of sample. Add drying agent. Transfer sample to extraction cell. If there is no sample available to perform a matrix spike/matrix spike duplicated a laboratory control spike duplicate must be performed.

11.2.9 Fill the void in each of the extraction cells with clean sand. (Dionex Operator's Manual 3-6.3).

11.2.10 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 1.0 mL of the 8270 surrogate standard solution by using a 1.0 ml syringe. In addition, add 1.0 mL 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.2.11 Attach the other cap to the other end of the extractor cell, making sure the tube is sitting flush on a hard surface so that no particulates get caught in the threads of the tube cap.

11.2.12 Place the extractor tube on the Dionex ASE 200 on a selected position with the serial number engraved end up on the top wheel. Place an empty 60 mL VOA collection vial, labeled with the sample ID, on the matching position on the bottom wheel. Schedule the Dionex ASE 200 and begin the cycle.

11.2.13 The Dionex ASE 200 extraction cycle:

Oven temperature: 100 º C Pressure: 1500 psi
Prepurge time: 0 minutes
Static time: 5 minutes
Heat: 5 minutes
Flush volume: 60%
Nitrogen purge: 60 sec. At 150 psi
Solvent A: 100%
Method rinse: ON
Static Cycles: 1
Extraction Fluid: (1:1) methylene chloride : acetone

11.2.14 Refer to section 11.4 for sample concentration.

11.3 Waste Dilution Extraction (SW846-3580)

11.3.1 (Refer to SOP FO-10 for subsampling guidance). Samples consisting of multiphase separations.

11.3.2 Pre-rinse "Class A" 10 ml volumetric with Methylene chloride.

11.3.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD).

11.3.4 Place the 10 ml volumetric on analytical balance (capable of accurately recording weight to the 0.01 g). Using a Pasteur pipet, transfer 1.0 g (to the nearest 0.1 g) to the volumetric. Record the weight.

11.3.5 Fill the volumetric half way with methylene chloride.

11.3.6 To the method blank, matrix spike/matrix spike duplicate, laboratory control spike, and all samples add 1.0 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

11.3.7 Bring samples up to volume with methylene chloride and cap for storage.

11.3.8 Add 2.0 grams of conditioned sodium sulfate to a 15ml amber vial with a Teflon cap. Transfer sample from the 10 ml volumetric flask to the 15ml vial.

11.3.9 Shake sample for two minutes.

11.3.10 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extracts through the glass wool and collect 5ml of the extract in a tube or vial.

11.3.11 No concentration step is need for this extraction. Sample will potentially require cleanup prior to analysis. Refer to attachment 1, 2, and 3 for sample cleanup options.

11.4 **Sample Concentration**

11.4.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds of the depth with Na_2SO_4. Rinse filter paper, Na_2SO_4, funnel, K-D apparatus, and concentrator tube with methylene chloride.

11.4.2 Quantitatively pour the extract through the filter and funnel seated on a concentrating, 500 ml Kuderna-Danish (K-D) for water samples or a 250mL Kuderna-Danish (K-D) for soil samples, apparatus complete with concentrator tube. Rinse the beaker three times with methylene chloride. Add these rinses through the filter and funnel into the K-D apparatus. Add a boiling chip to the K-D flask prior to placing it on the heated water bath. Wet a three ball Snyder column with approximately 2 mL of methylene chloride. Attach the Snyder column.

11.4.3 Place the K-D in the heated water bath so the concentrator tube is immersed in the water and the lower rounded surface of the K-D is bathed in steam. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood (set the knob of the temperature control to 4~5). It is critical that the analyst watch the extract as it distills. THE EXTRACT MUST NOT GO TO DRYNESS.

11.4.4 When the extract volume reaches approximately 5-7 mL, remove the K-D from the bath. Slightly tilt the apparatus and rotate to aid in solvent drainage from the Snyder column. Allow it to cool completely.

11.4.5 Remove the Snyder column, rinse the ground glass joints with a small amount of methylene chloride and then remove the K-D flask. Turn on the heating unit for the Organomation. The water bath shall be about 35ºC. Place sample concentrator tube into the nitrogen blow down apparatus. Allow a gentle stream of nitrogen to interact with the extract. There shall be no splashing or excessive movement upon the surface of the extract. Allow the extract to evaporate down

to 0.8 ml. Remove concentrator tube from water bath and by using a Pasteur pipet, bring sample extract up to 1.0 ml volume with methylene chloride.

11.4.6 Transfer the 1 mL of the extract to a labeled amber screw-cap injection vial. Record the final extract on the injection extraction bench sheet.

11.4.7 The sample extract is now ready for analysis. If samples are not analyzed immediately store the sample extract in a freezer.

11.4.8 Sample will potentially require cleanup prior to analysis. Refer to attachments I, II, or III for sample cleanup options.

12.0 SAMPLE ANALYSIS

12.1 Sample Sequence

12.1.1 It is highly recommended that sample extracts be screened on a GC/FID to protect the GC/MS system from unexpectedly high concentrations of organic compounds.

12.1.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 ul of the internal standard solution to the 1-ml concentred sample extract obtained from sample preparation. Alternatively, 2 uL of internal standard solution is added to 0.2 mL of sample extract in a vial insert.

12.1.3 Before initial calibration or sample analysis a priming standard can be injected at a level up to twice the highest linearity point.

12.1.4 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so a 25 ng injection of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 3. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and Pentachlorophenol shall be present at their normal responses and peak tailing evaluated. Benzidine and Pentachlorophenol must each have tailing factors less than 2. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

12.1.5 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing target analytes, prior to conducting any sample analysis. A CCV must be injected at the begining of each twelve hour shift. The SPCCs must meet minimum response factor of 0.050. The percent difference of the CCCs must be less than or equal to 20%. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that are analyzed by this method, it is expected that some compounds will fail to meet the criterion. In cases where compounds fail, they can still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations where the failed compound is present, the concentrations must be reported as estimated values.

12.1.6 The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the extracted ion chromatographic profile area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the CCV level from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCVs and associated samples while the system was malfunctioning is necessary. The retention times and standard reference spectra in the method are updated from the CCV for each 12 hour sequence.

12.1.7 Samples can be directly injected after the successful analyses of the initial calibration curve, ICV, DFTPP, and CCV. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries shall be compared to laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections. Some sample extracts will potentially require clean-up procedures. Refer to attachments I, II and III.

12.1.8 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS, the sample extract must be diluted and reanalyzed. Additional internal standards must be added to the diluted extract to maintain the same concentration as in the calibration standards.

12.1.9 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method (SW-846-8270). The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

12.1.9.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

12.1.9.2 The relative retention time (RRT) of each compound in each calibration standard agree within 0.06 RRT units.

12.1.9.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.1.9.4 Structural isomers that produce very similar spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two

isomeric peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs that are separable by the GC are identified, quantitated and reported as the sum of both compounds by the GC.

12.1.10 For samples containing components not associated with the calibration standards, a library search can be made for the purpose of tentative identification. Data system libraries search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

12.1.11 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the extracted ion chromatographic profile.

12.1.12 Where applicable, the concentration of any non-target analytes identified in the sample shall be estimated. The same formulas that are used for targe comounds are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms, and the RF for the compound is assumed to be one.

12.1.13 The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

12.2 Sample Calculations

12.2.1 Re-arranging the equation from sec. 10.1.5 to calculate the "as-analyzed" value yields:

$$C_s = \frac{A_s \times C_{is}}{A_{is} \times RF}$$

RF = Average Response Factor
A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate in ug/mL.
C_{is} = Concentration of the internal standard in ug/mL.

12.2.2 Once the target components of the extract have been identified and quantitated, the "as-analyzed" value is converted to the "as-received" concentration as follows:

Water Matrix:

$$\frac{(ug/mL\ injected)\,x\,(mL\ extract\ \ final\ volume)x(dilutionfactor)}{(volume\ of\ \ sample\ extracted, in\ L)} = \mu g/L$$

Soil Matrix:

$$\frac{(ug/mL\ injected)\,x\,(mL\ extract\ \ final\ volume)x(dilutionfactor)}{(weight\ of\ \ sample\ extracted, in\ g)} = ug/g$$

13.0 Data Assessment & Acceptance Criteria for QC Measures.

13.1 Data Assessment & Acceptance Criteria for QC Measures.

13.1.1 If the initial analysis of a sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be re-analyzed at a dilution.

13.1.2 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method SW-846-8270.

13.1.3 The relative retention time (RRT) of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

13.1.4 Sporadic Marginal Failures: The number of target analytes reported for the method will dictate the number of allowable QC failures in spikes (LCS, MS/MSD) as given below. The sporadic marginal failure allowance entails the application of an expanded acceptance criterion.

Table 3.1
Sporadic Marginal Failures

N[1]	X[2]
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

13.2 Reporting Quantitative Analysis

13.2.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times and mass ions are compared to the sample retention times, the sample data can be reported.

13.2.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 5.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs the checklist. Senior analysts have the authority to validate their own data. Finally, the validated results are made available to the client services personnel.

13.2.3 An electronic copy of the data is then filed or archived. The package includes; the sequence run log, the LIMS run log, verification of calibration data and chromatograms/quant reports. All the data is to be initialed and dated by the

analyst. Each data file header contains the Prep ID #, date prepped, Analytical ID #, and date of sequence.

13.3 Corrective Measures for Out-of-Control Data

13.3.1 When data is out of control, corrective actions are required. Certain situations will cause data deficiencies, for example: if the non-conformities involve failing QC within the analytical sequence batch, the reanalysis of samples can eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data will correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument will be cleaned and decontaminated, and the sample will be diluted or undergo sample clean-up procedures to reduce matrix effect. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions are to be taken for different QC-type failures or non-conformities (Table 4.)

13.4 Contingencies for Handling Out-of-Control or Unacceptable Data

13.4.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data has the potential to be out of control or unacceptable to report. Since these instances can arise, contingency plans need to be in place to prevent and/or minimize their effect on data.

13.4.2 To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises re-analysis or re-extraction is an option.

13.4.3 If a sample is extracted passed holding times, the analyst must notify client services immediately. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur. If the client still request sample analysis, the sample data is qualified for holding time violations.

13.4.4 If the non-conformities involve failing QC within the analytical sequence batch, the analyst must reanalyze the extract. This will ensure that the instrument was functioning properly. If the results are passing, the non-conformities were associated with the instrument. If data is still outside QC criteria, re-extraction of the all samples associated with the QC samples must be performed (provided that there is sample available). If no sample is available for re-extraction, the samples are qualified accordingly.

13.4.5 If the out of control data is the result of instrument malfunctions, the analyst must evaluated and repair instrument prior to reanalysis of the affected data. After the instrument maintenance and repair of the downed instrument which caused data to be out of control must demonstrate corrective measures by analyzing a Continuing Calibration Verification (CCV) before continuing sample analysis. Any maintenance and repair must be noted in the instrument logbook.

13.4.6 If sample matrix affect or contamination is the reason for poor data, the analyst

will use professional judgement for the reanalysis of the extract. The extract can be diluted and reanalyzed. The sample can be re-extracted confirming matrix interference. The analyst can report and qualify the sample data. The analyst must supply explanation for the qualified data in the narrative or client notes.

13.4.7 If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

13.4.8 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

13.4.8.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

13.4.8.2 In all instances, if results are reported from data that is out of control or unacceptable, that data is qualified accordingly. Once the client has been notified and he or she instructs us to report the data, then flag the data indicating what type of nonconformity has occurred.

13.4.8.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data.

13.4.8.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

14.0 WASTE MANAGEMENT

14.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

ADDENDUM

Tentatively Identified Compounds:

For samples containing compounds not associated with the calibration, a library search of the NBS75K library may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Up to 10 organic compounds of greatest apparent concentration shall be reported. Compounds with responses less than 10 percent of the nearest internal standard shall not be reported.

Only after visual comparison of sample spectra with the nearest library searches may the analysts assign a tentative identification. When analysts are requested to identify the TICs, the following guidelines will be used (see SW846 methods 8260 and 8270):

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within $\pm$ 20% ($\pm$ 30% for 8270). (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Where applicable, the concentration of any non-target analytes identified in the sample should be estimated. The same formulae that are used for target compounds should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound will be assumed to be 1.

ATTACHMENT I
GEL-PERMEATION CLEANUP
METHOD 3640A

1.0 SCOPE AND APPLICATION

1.1 Gel-Permeation Chromatography (GPC) is a size exclusion cleanup procedure using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and is characterized by the range or uniformity (exclusion range) of that pore size. In the choice of gels, the exclusion range must be larger than the molecular size of the molecules to be separated. A cross-linked divinylbenzene-styrene copolymer (SX-3 Bio Beads or equivalent) is specified for this method.

1.2 General cleanup application – GPC is recommended for the elimination from the sample of lipids, polymers, copolymers, proteins, natural resins and polymers, cellular components, viruses, steroids, and dispersed high-molecular-weight compounds. GPC is appropriate for both polar and non-polar analytes; therefore, it can be effectively used to cleanup extracts containing a broad range of analytes.

1.3 Normally, this method is most efficient for removing high boiling materials that condense in the injection port area of a gas chromatograph (GC) or in the front of the GC column. This residue will ultimately reduce the chromatographic separation efficiency or column capacity because of adsorption of the target analytes on the active sites. Pentachlorophenol is especially susceptible to this problem. GPC, operating on the principal of size exclusion, will not usually remove interference peaks that appear in the chromatogram since the molecular size of these compounds is relative similar to the target analytes. Separation cleanup techniques, based on other molecular characteristics (i.e. polarity), must be used to eliminate this type of interference.

1.4 This method is restricted for use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method is used to cleanup extracted samples with unwanted light and/or heavy compounds that interfere with final analysis of methods; 8081, 8082, 8270 and 8310.

2.2 Samples are extracted then concentrated and prepared for GPC cleanup.

2.3 Determinations for which samples that are candidates for GPC cleanup are based on client specific, site specific and project specific requirements, specific

characteristics of initial extract (e.g. color or odor) or interference determined by initial extract analysis.

2.4 The column is calibrated and then loaded with the sample extract to be cleaned up. Elution is effected with a suitable solvent(s) and collection times are adjusted based on desired analysis. The collected fractions are then concentrated per guidelines for methods; 8081, 8082, 8270 and 8310.

3.0 INTERFERENCES

3.1 A reagent blank is to be analyzed for the compound of interest prior to the use of this method. The level of interferences must be below the estimated quantitation limits (EQL's) of the analytes of interest before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method will be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Gel-permeation chromatography system - Gilson GV-271 ASPEC, Gilson, INC or equivalent. All systems, whether automated or manual must meet the calibration requirements.

4.2 Chromatographic column -350mm x 21.20 mm 0 micron, Phenomenex p/n 00W-3035-PO or equivalent.

4.3 Guard column – (optional) 60mm x 21.20 mm 0 micron, Phenomenex p/n 03R-3035-PO or equivalent.

4.4 Ultraviolet detector –fixed wavelength (254 nm) with a semi-prep flow-through cell, Gilson 112 UV/VIS detector, Gilson, INC..

4.5 Strip chart recorder, recording integrator or laboratory data system, (Trilution and LIMS).

4.6 Syringe – 10ml with Luerlok fitting.

4.7 Syringe filter assembly, disposable – Puradisc 25TF sample filter assembly Whatman #6784-2510, 25mm and 1.0 micron filter discs or equivalent. Check each batch for contaminants. Rinse each filter assembly (prior to use) with methylene chloride if necessary.

4.8 Analytical balance – 0.00 g.

4.9 Volumetric flasks, Class A 5ml to 100ml

4.10 Graduated cylinders

4.11 Disposable glass culture tubes 13mm x 100mm, (Kimble 73500-13100).

4.12 Disposable glass collection tubes- (Kimax 51 25mm x 200mm, Kimble 45060-25200).

4.13 Pasteur pipets- (Corning #7095B-5X)

Reagents

4.14 Methylene chloride, Pesticide grade.

4.15 Cyclohexane, Pesticide grade.

4.16 N-Butyl chloride, Pesticide grade.

5.0 CALIBRATION

5.1 Preparation of standards is documented in the GPC standard logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards once opened expire within six months or sooner if routine QC indicates a problem and not to exceed the manufactures expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire in six months and not to exceed the opened date of the stock standard and not to exceed manufactures expiration date.

5.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufactures suggested expiration date. Stock standards once opened expire in six months and not to exceed the manufactures expiration date.

5.3 The following is the stock standard that is commercially prepared standard, which is certified by the manufacturer;

GPC Calibration Mix: Restek Part # 32019. (1ml ampul)
GPC Calibration Mix: Restek Part # 32023. (5ml ampul)

5.4 GPC Calibration Solution is a certified prepared standard in methylene chloride containing the following analytes (in elution order):

Compound	mg/mL
Corn oil	250

Bis (2-ethylhexyl) phthalate	10
Methoxychlor	2.0
Perylene	0.2
Sulfur	0.8

Table 1.0 GPC Calibration Standard

Standard	Component Name	Conc. (mg/ml)	STD Volume (ml)	Final Volume (ml)	Final Conc. (ug/ml)
GPC STD	Corn Oil	250	5.0	50.0	25000.0
	Bis(2-ethylhexyl)pht.	10	5.0	50.0	1000.0
	Methoxychlor	2.0	5.0	50.0	200.0
	Perylene	0.2	5.0	50.0	20.0
	Sulfur	0.8	5.0	50.0	80.0

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND EXTRACTION PROCEDURES

6.1 Follow guidelines listed in section 8.0 in SOP 8081 Rev. 5 for sample collection, preservation and handling.

7.0 PROCEDURE

7.1 It is very important to have consistent laboratory temperatures during an entire GPC run, which could be 24 hours or more. If temperatures are not consistent, retention times will shift and the dump and collect times determined by the calibration standard will no longer be appropriate. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 72°F.

7.2 **GPC Setup**

Column Preparation:

7.2.1 Using manual control options in Trilution set the system to equilibrate for at least 30 minutes prior to starting a run or priming.

7.2.2 If system has not been used on a regular basis, prime all lines and pumps either manually or using the automated system following guidelines listed in the Trilution help manual.

7.3 **Calibration of the GPC Column**

7.3.1 Place approximately 6 to 7 mls of the calibration solution in a disposable culture tube. Place tube in position 1 on the sample tray. Set Trilution to run a calibration method with an inject volume of 5.50ml.

7.3.2 Following are criteria for evaluation of the UV chromatogram for column condition.

7.3.3 Peaks must be observed and will be symmetrical for all compounds in the calibration solution.

7.3.4 Corn oil and phthalate peaks must exhibit >85% resolution.

7.3.5 Phthalate and methoxychlor peaks must exhibit >85% resolution.

7.3.6 Methoxychlor and perylene peaks must exhibit >85% resolution.

7.3.7 Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

7.3.8 Nitroaromatic compounds are particularly prone to adsorption. For example 4-nitrophenol recoveries can be low due to a portion of the analyte being discarded after the end of collection time. Columns are to be tested with the semivolatiles matrix spiking solution. GPC elution will continue until after perylene has eluted or long enough to recover at least 85% of the analytes, whichever time is longer.

7.3.9 Calibration for Semivolatiles – Using the information from the UV trace, establish appropriate collect and dump time periods to ensure collection of all target analytes. Initiate column eluate collection just before elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Collection can be stopped before sulfur elutes. Each laboratory is required to establish its specific time sequences.

7.3.10 Calibration for Organochlorine Pesticides/PCBs – Determining the elution times for the phthalate, methoxychlor, perylene and sulfur. Choose a dump time which removes >85% of the phthalate, but collects >95% of the methoxychlor. Stop collection after the elution of perylene, but before sulfur elutes.

7.3.11 Calibration for Polynuclear Aromatic Hydrocarbons – Determine the elution times for corn oil, phthalate and perylene. Start elution collections just before the elution of bis (2-ethylhexyl) phthalate and end collection after the elution of perylene.

7.3.12 Verify the flow rate by collection column eluate for 10 minutes in a graduated cylinder and measure the volume which will between 45-55ml (4.5-5.5 ml/min). If flow rate is outside of this range, corrective action must be taken, as described above. Once flow rate is within the ranges of 4.5-5.5 ml/min, record the column pressure (6-10 psi) and room temperature. Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If

flow rate and/or column pressure do not fall within the above ranges the column or pre-column are suspected. A UV trace that does not meet the criteria in section 7.2.2.2 would also indicate that the column or pre-column are responsible the poor performance.

7.3.13 Reinject the calibration solution after appropriate collect and dump cycles have been set and the solvent flow and column pressure have been established.

7.3.14 Measure and record the volume of collected GPC eluate in a graduated cylinder. The volume of GPC eluate collected for each sample extract processed can be used to indicate problems with the system during sample processing.

7.3.15 The retention times for bis (2-ethylhexyl) phthalate and perylene must not vary more than ± 5% between calibrations. If the retention time shift is >5%, take corrective action. Excessive retention time shifts are caused by:

7.3.15.1 Poor laboratory temperature control or system leaks.

7.3.15.2 An unstabilized column that requires pumping methylene chloride through it for several more hours.

7.3.15.3 Excessive laboratory temperatures, causing outgassing of the methylene chloride.

7.3.16 Analyze a GPC blank by loading 5 ml of methylene chloride into the GPC. Concentrate the methylene chloride that passes through the system during the collect cycle using a Kuderna-Danish (KD) evaporator. Analyze the concentrate by whatever detectors will be used for the analysis of future samples. Exchange the solvent if necessary. If the blank exceeds the estimated quantitation limit of the analytes, pump additional methylene chloride through the system for 1 to 2 hours. Analyze another GPC blank to ensure the system is sufficiently clean. Repeat the methylene chloride pumping if necessary.

7.3.17 A hard copy calibration report will follow the samples in the sample prep folder.

8.0 EXTRACT PREPARATION

8.1 Adjust the extract volume to 5 ml. The solvent extract must be primarily methylene chloride. All other solvents, e.g. 1:1 methylene chloride/acetone, must be concentrated to 1 ml (or as low as possible if a precipitate forms) and diluted to 5 ml with methylene chloride. Thoroughly mix the extract before proceeding.

8.2 Filter the extract through a 1 micron filter disc by attaching a syringe filter assembly containing the filter disc to a 10 ml syringe. Draw the sample extract through the filter assembly and into the 10 ml syringe. Disconnect the filter

assembly before transferring the sample extract into a small glass container, e.g. a 15 ml culture tube with a Teflon lined screw cap. Alternatively, draw the extract into a syringe without the filter assembly, attach the filter assembly and force the extract through the filter and into the glass container. The latter is the preferred technique for viscous extracts or extracts with a lot of solids. Particulate larger than 5 microns can scratch the valve, which can result in a system leak and cross-contamination of sample extracts in the sample loops.

NOTE**:** Viscosity of a sample extract can not exceed the viscosity of 1:1 water/glycerol. Dilute samples that exceed the viscosity.

9.0 SCREENING THE EXTRACT

9.1 Screen the extract to determine the weight of dissolved residue by evaporating a 100 µL aliquot to dryness and weighing the residue. The weight of dissolved residue loaded on the GPC column cannot exceed 0.500 g. Residues exceeding 0.500 g will very likely result in incomplete extract cleanup and contamination of the GPC switching valve (which results in cross-contamination of sample extracts).

9.1.1 Transfer 100µL of the filtered extract from section 8.2 to a tared aluminum weighing dish.

9.1.2 A suggested evaporation technique is to use a heat lamp. Set up a 250 watt heat lamp in a hood so that it is 8 ± 0.5cm from a surface covered with a clean sheet of aluminum foil. Surface temperature will be between 80-100°C (check temperature by placing a thermometer on the foil and under the lamp). Place the weighing dish under the lamp using tongs. Allow it to stay under the lamp for 1 minute. Transfer the weighing dish to an analytical balance or a micro balance and weigh to the nearest 0.1mg. If the residue weight is less than 10mg/100µL, then further weighings are not necessary. If the residue weight is greater than 10mg/100µL then determine if constant weight has been achieved by placing the weighing dish and residue back under the heat lamp for 2 or more additional 0.5 minute intervals. Reweigh after each interval. Constant weight is achieved when three weights agree within ±10%.

9.1.3 Repeat the above residue analysis on a blank and a spike. Add 100µL of the same methylene chloride used for the sample extraction to a weighing dish and determine residue as above. Add 100µL of a corn oil spike (5g/100ml) to another weighing dish and repeat the residue determination.

9.1.4 A residue weight of 10mg/100µL of extract represents 500mg in 5ml of extract. Any sample extracts that exceed the 10mg/100µL residue weight must be diluted so that the 5ml loaded on the GPC column does not exceed 0.500g. Following is a calculation that will be used to determine what dilution is necessary if the residue exceeds 10mg.

Y ml taken for dilution = 5 ml final volume x $\frac{\text{10mg maximum}}{\text{X mg of residue}}$

Example

Y ml taken for dilution = 5 ml final volume x $\frac{\text{10mg maximum}}{\text{15 mg of residue}}$

Y ml taken for dilution = 3.3 ml

Therefore, taking 3.3 ml of sample extract from 7.4.2 and diluting it to 5ml with methylene chloride will result in 5 ml of diluted extract loaded on the GPC column that contains 0.500 g of residue.

NOTE**:** This dilution factor must be included in the final calculation of analyte concentrations.

10.0 GPC CLEANUP

10.1 Calibrate the GPC at least once per week following the procedure outlined in section 7.3. Ensure that UV trace requirements, flow rate and column pressure criteria are acceptable. Also, retention time shift must be <5% when compared to retention times in the last calibration UV trace.

10.2 If these criteria are not met, follow appropriate maintenance methods to try to regain resolution.

10.3 Load sample vials with 5 ml of filtered extract from section 7.4.2. The Method Blank (MB) and Laboratory Control Standard (LCS) must be run through the GPC process from each prep batch that the samples originated from.

NOTE**:** The number of samples from each prep batch that are put through GPC will vary by client, location and project specifications

10.4 Set the GPC up to run cleanup of the samples, verifying that collection and dump times correlate with the calibration times for each analysis specified in section 7.3. Multiple methods can be used on each GPC run. Follow software specific guidelines for setting up a GPC run.

NOTE**:** It will be necessary to run multiple rinse methods between samples to ensure no cross-contamination between particularly dirty samples

10.5 Monitor sample volumes collected. Changes in sample volumes collected will indicate one or more of the following problems:

10.5.1 Change in solvent flow rate caused by channeling in column or changes in column pressure.

10.5.2 Increase in column operating pressure due to the absorption of particles or gel fines onto either the guard column or the analytical column gel, if a guard column is not used.

10.5.3 Leaks in the system or significant variances in room temperature.

10.6 Concentrate the extract by following the methods listed in the sections for Sample Concentration for methods; 8081, 8082, 8270 and 8310.

11.0 QUALITY CONTROL

11.1 The analyst will demonstrate that the compound(s) of interest are being quantitatively recovered before applying this method to actual samples.

11.2 For sample extracts that are cleaned up using this method, the associated quality control samples must also be processed through this clean up method.

11.3 Some brands of methylene chloride can contain unacceptably high levels of acid (HCl). Check the pH by shaking equal portions of methylene chloride and water, and then check the pH of the water layer.

11.4 If the pH of the water layer is ≤ 5, filter the entire supply of solvent through a 2 in x 15 in glass column containing activated basic alumina. This column will be sufficient for processing approximately 20-30 liters of solvent. Alternatively, find a different supply of methylene chloride.

ATTACHMENT II
ACID-BASE PARTITION CLEANUP
METHOD 3650B

1.0 SCOPE AND APPLICATION

1.1 Method 3650 is a liquid/liquid partitioning cleanup method to separate acid analytes from base/neutral analytes using pH adjustments. It can be used for cleanup of petroleum waste prior to analysis or futher cleanup.

Compounds by Fraction

Compound	Fraction
Benzo (a) anthracene	Base-Neutral
Benzo (a) pyrene	Base-Neutral
Benzo (b) fluoranthene	Base-Neutral
Chlordane	Base-Neutral
2-Chlorophenol	Acid
Chrysene	Base-Neutral
Cresol(s)	Acid
Dichlorobenzene(s)	Base-Neutral
2,4-Dimethylphenol	Acid
Dinitrobenzene	Base-Neutral
4,6-Dinitro-2-Methylphenol	Acid
2,4-Dinitrotoluene	Base-Neutral
Hexachlorobenzene	Base-Neutral
Hexachlorocyclopentadiene	Base-Neutral
Naphthalene	Base-Neutral
Nitrobenzene	Base-Neutral
4-Nitrophenol	Acid
Pentachlorophenol	Acid
Phenol	Acid
Pyridine	Base-Neutral
Tetrachlorobenzene(s)	Base-Neutral
Tetrochlorophenols(s)	Acid
Trichlorophenol(s)	Acid

1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The solvent extract from a prior extraction method is shaken with water that is strongly basic. The acid analytes partition into the aqueous layer, whereas, the basic and neutral compounds stay in the organic solvent. The base/neutral fraction is concentrated and is then ready for further cleanup, if necessary, or analysis. The aqueous layer is acidified and extracted with an organic solvent. This extact is concentrated and is then ready for analysis of the acid analytes.

3.0 INTERFERENCES

3.1 More extensive procedures than those outlined in this method are sometimes

necessary for reagent purification.

3.2 A method blank must be run for the compounds of interest prior to use of the method. The interferences must be below the method detection limit before this method is applied to actual samples.

4.0 APPARATUS AND MATERIALS

4.1 Drying column –20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom, or equivalent.

4.2 Autosampler vials.

4.3 Heated water bath.

4.4 Boiling Chips.

4.5 pH paper (range 0-14).

4.6 Separatory Funnel.

4.7 Erlenmeyer Flask, 125 ml.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades can be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water.

5.3 Sodium Hydroxide, (10N)

5.4 Sulfuric Acid, (1:1 v/v in water)

5.5 Sodium Sulfate (Purified by heating to 400º).

5.6 Methylene Chloride, pesticide grade.
5.7 Acetone, pesticide grade.

5.8 Methanol, pesticide grade.

5.9 Diethyl Ether, pesticide grade.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to Method 8270, Section 8.0 for sample handling.

7.0 PROCEDURE

7.1 Place 10 ml of solvent extract from a prior extraction procedure into a 125 ml separatory funnel.

7.2 Add 20 ml of methylene chloride to the separatory funnel.

7.3 Slowly add 20 ml of prechilled organic-free reagent water which has been previously adjusted to a pH of 12-13 with 10N sodium hydroxide.

7.4 Seal and shake the separatory funnel for at least 2 minutes with periodic venting to release excess pressure.

7.5 Allow the organic layer to separate from the aqueous phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and can include stirring, filtration, centrifugation, or other physical methods.

7.6 Separate the aqueous phase and transfer it to a 125 ml Erlenmeyer flask. Repeat the extraction two more times using 20 ml aliquots of dilute sodium hydroxide. Combine the aqueous extracts.

7.7 Water soluble organic acids and phenols will be primarily in the aqueous phase. Base/Neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase, discard the methylene chloride and proceed to Section 7.8. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 7.10.

7.8 Externally cool the 125 ml Erlenmeyer flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Quantitatively transfer the cool aqueous phase to a clean 125 ml separatory funnel. Add 20 ml of methylene chloride to the separatory funnel and shake for at least 2 minutes. Allow the methylene chloride to separate from the aqueous phase and collect the methylene chloride in an Erlenmeyer flask.

7.9 Add 20 ml of methylene chloride to the separatory funnel and extract at pH 1-2 a second time. Perform a third extraction in the same manner combining the extracts in the Erlenmeyer flask.

7.10 Prepare the extracts for sample concentration. Refer to method 8270, Section 9.3 for sample concentration techniques.

8.0 QUALITY CONTROL

8.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples.

8.2 For samples that are cleaned using this method, the associated quality control samples must be processed through this cleanup method.

ATTACHMENT III
FLORISIL CARTRIDGE CLEANUP
METHOD 3620B

1.0 SCOPE AND APPLICATION

1.1 Florisil, a registered trade name of U. S. Silica Co., is a magnesium silicate with basic properties. It is used to separate analytes from interfering compounds prior to sample analysis by a chromatographic method.

1.2 Florisil has been used for the cleanup of pesticide residues and other chlorinated hydrocarbons; the separation of nitrogen compounds from hydrocarbons; the separation of aromatic compounds from aliphatic-aromatic mixtures; and similar applications for use with fats, oils, and waxes. Additionally, Florisil is considered good for separations with steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates. Florisil cleanup can be accomplished using a glass chromatographic column packed with Florisil or using solid-phase extraction cartridges containing Florisil. This method includes procedures for cleanup of sample extracts containing the following analyte groups:

Chlorinated hydrocarbons
Organochlorine pesticides
Organophosphates
Organophosphorus pesticides
PCBs
Phthalate esters
Nitrosamines
Nitroaromatics
Haloethers
Aniline and aniline derivatives

2.0 SUMMARY OF METHOD

2.1 This method describes procedures for Florisil cleanup of solvent extracts of environmental samples using solid-phase extraction cartridges. The cartridge cleanup protocol uses solid-phase extraction cartridges containing 40 µm particles of Florisil (60 D pores). Each cartridge is washed with solvent immediately prior to use. The sample extract is loaded onto the cartridge which is then eluted with suitable solvent(s). A vacuum manifold is required to obtain reproducible results. The eluate can be further concentrated prior to gas chromatographic analysis.

3.0 INTERFERENCES

3.1 A reagent blank is prepared and analyzed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples. The procedures for reagent purification outlined here are considered to be the minimum requirements for use of this method. More extensive procedures are sometimes necessary to achieve acceptable levels of interferences for some analytes. However, during the evaluation of the cartridge clean-up procedure, phthalate esters were detected in the Florisil cartridge method blanks at concentrations up to 400 ng per cartridge. Therefore, complete removal of the phthalates esters from Florisil cartridges is not possible.

4.0 APPARATUS AND MATERIALS

4.1 Vacuum manifold - VacElute Manifold SPS-24 (Analytichem International), Visiprep (Supelco, Inc.) or equivalent, consisting of glass vacuum basin, collection rack and funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500-mL sidearm flask fitted with a one-hole stopper and glass tubing. The manifold is required for use of the cartridge cleanup protocol.

5.0 REAGENTS

5.1 Eluting solvents - all solvents must be pesticide quality or equivalent.

5.2 Hexane, pesticide grade.

5.3 Methylene chloride, pesticide grade.

5.4 Acetone, pesticide grade.

5.5 Pentane, pesticide grade

5.6 Ethyl Ether, pesticide grade

5.7 Florisil cartridge phenol check solution (for the organochlorine pesticide technique) - Prepare a solution of 2,4,5-Trichlorophenol in acetone at a concentration of 0.1 g/L.

5.8 Florisil cartridge pesticide check solution - Prepare a solution containing the following analytes in hexane:

BHCs
Heptachlor
BHC
Endosulfan I
Dieldrin
Endrin
4,4'-DDD
4,4'-DDT
Methoxychlor
Tetrachloro-m-xylene
Decachlorobiphenyl

In addition to the pesticide cartridge check, target analytes for any given method are subject to evaluation by adding 1 ml of CCV standard to the Florisil cartridge for testing.

6.0 PROCEDURE

6.1.1 Whenever Florisil is used to fractionate groups of target compounds (rather than to simply remove potential interference) it is critical that the specific fractionation scheme be validated using spiked solutions or spiked sample

extracts that contain most or all of the analytes of interest. This is particularly important when the Florisil cartridge techniques are employed, as the differences between the various cartridge formats and manufacturers affect the fractionation patterns. In addition, it is useful to archive any fractions not originally intended for analysis, in the event that the fractionation scheme chosen does not yield the intended results. Once the determinative analysis has been performed and demonstrates that the fractionation has been successful, such archived fractions are disposed of in an appropriate manner. However, if the fractionation did not perform as intended, the analytes of interest can be contained in the archived fractions, which can be analyzed or combined with the other fraction(s) for reanalysis.

6.1.2 Following Florisil cleanup, extracts can require further concentration and/or solvent exchange.

6.2 Cartridge Evaluation

6.2.1 The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered can be used to process the samples. A check is also performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

6.2.2 To check each new lot of Florisil cartridges before use, perform the following in duplicate.

6.2.3 Combine 0.5 ml of 2,4,5-Trichlorophenol solution in Section 5.5 and 0.5 ml of pesticide spiking solution in Section 5.6 in a two ml autosampler vial.

6.2.4 Arrange the cartridges on the manifold in the closed-valve position.

6.2.5 Turn on the vacuum pump and set the vacuum to 10 in (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates are controlled by opening and closing cartridge valves.

6.2.6 Condition the cartridges by adding 4 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

6.2.7 Slowly open cartridge valves to allow the hexane to pass through the cartridges.

6.2.8 Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed.

6.2.9 Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

6.2.10 Add the spiking solution in 6.2.3 to the Florisil cartridge.

6.2.11 Elute the cartridge with 9 ml of acetone/hexane (10/90, v/v). Collect the extraction in a 15 ml test tube.

6.2.12 Transfer extract to a concentrating thimble. Using the nitrogen blow down apparatus, concentrate the extract down to one ml and analyze by Method 8081A.

6.2.13 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110%, if the trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

6.3 Handling sample extracts

6.3.1 Most sample extracts will have to be concentrated to a smaller volume prior to the use of Florisil cleanup. The extract volume is a function of the analytical sensitivity necessary to meet the project objectives. The extract volume will also affect the ability of the Florisil to separate target analytes from potential interferences where applying large extract volumes to the cartridges can cause poor results. Consult the appropriate extraction and determinative methods for the details on final extract volumes, extract concentration techniques, and solvent exchange procedures.

6.3.2 Place Florisil cartridges on manifold. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Condition the cartridges by adding 4 ml of ethyl ether/pentane (15/85, v/v). Slowly open the cartridge valve and collect the eluate into the collection vial. Allow a few drops of ethyl ether/pentane (15/85, v/v) to pass through the cartridge to remove any air bubbles. Close valve and allow solvent to soak the entire sorbent bed for five minutes. Do not turn off vacuum.

6.3.3 Slowly, open the valve to allow the ethyl ether/pentane (15/85, v/v) to pass through the cartridge. Close valve when about 1 mm of ethyl ether/pentane (15/85, v/v) is remaining above the sorbent bed.

6.3.4 Quantitatively transfer the 1 ml extract into the cartridge using an additional 2 ml of pentane to complete the transfer.

6.3.5 Just prior to the exposure of the Florisil layer to the air, elute the column with 10 ml of ethyl ether/pentane (15/85, v/v). Discard the eluate. This fraction will contain diphenylamine.

6.3.6 Elute the column with 5 ml of acetone/ethyl ether (5/95, v/v) collecting the eluate in a collection vial. This fraction will contain all of the nitrosamines listed in the scope of the method SW846-8270.

6.3.7 Close valve when about 1 mm of acetone/ethyl ether (5/95, v/v) is remaining above the sorbent bed.

6.3.8 Before extract goes below the surface of the sorbent bed, add 9 ml of acetone/ethyl ether (5/95, v/v) to the cartridge.

6.3.9 After extract has been collected, concentrate sample down to 1 ml. Refer to Section 9.3 for concentration techniques.

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6.3.10 As needed, perform a solvent exchange and adjust the final volume of the eluant to 1 ml.

7.0 QUALITY CONTROL

7.1 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentration. The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered can be used to process the samples. A check is also performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

7.2 To check each new lot of Florisil cartridges before use, perform the following in duplicate. Combine 0.5 mL of the 2,4,5-trichlorophenol solution, 1.0 mL of the pesticide solution, and 0.5 mL of hexane in a vial. Condition the cartridge and then perform the cartridge cleanup Elute the cartridge with 9 mL of acetone/hexane (10/90, v/v) only. Reduce the volume to 1.0 mL and analyze. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110 %, if the recovery of trichlorophenol is less than 5 %, and if no peaks interfering with the target analytes are detected. In addition to the pesticide cartridge check, target analytes for any given method are subject to evaluation by adding 1 ml of CCV standard to the Florisil cartridge for testing

ATTACHMENT IV
SILICA GEL CLEANUP
METHOD 3630C

1.0 SCOPE AND APPLICATION

1.1 Silica gel is a regenerative adsorbent of silica with weakly acidic properties. Silica gel can be used in column in column chromatography for the separation of analytes from interfering compounds of different chemical polarity.

1.2 This method is restricted to use by or under the supervision of trained analyst. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 REFERENCES

2.1 USEPA, SW-846, 3rd Ed. Method 3630C. December, 1996

3.0 SUMMARY OF METHOD

3.1 This method provides the option of using either standard column chromatography techniques or solid phase extraction techniques.

3.2 Cartridge clean-up protocol uses solid phase extraction cartridges packed with 1 gram of 2 grams of silica gel absorbent. Each cartridge is solvent washed immediately prior to use. Aliquots of sample extracts are loaded onto the cartridges, which are then eluted with suitable solvents. A vacuum manifold is required to obtain reproducible results. The collected fractions can be further concentrated prior to analysis.

4.0 APPARATUS AND MATERIALS

4.1 Silica Gel cartridges 1 gram or 2 grams. (100/200 mesh).

4.2 Vacuum manifold system.

4.3 Pipets – disposable, Pasteur type.

4.4 Collection vials, 20 ml tubes.

4.5 Autosample vials.

4.6 Cyclohexane, pest grade.

4.7 Hexane, pest grade.

4.8 Acetone, pest grade.

4.9 Pentane, pest grade.

4.10 2-Propanol, pest grade.

4.11 Toluene, pest grade.

4.12 Methylene Chloride, pest grade

5.0 PROCEDURE

5.1 Cartridge Conditioning

5.1.1 Arrange the 2 g silica cartridge on the manifold in the closed-valve position. Different gram size of silica cartridges can be used.

5.1.2 Turn on the vacuum pump and set pump vacuum to 10 inches (254 mm) of Hg.

5.1.3 Condition the cartridges by adding 4 ml of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off vacuum.

5.1.4 Slowly open cartridge valves to allow the hexane to pass through the cartridges. Close the cartridge valves when there is still at leasst 1 mm of solvent above the sorbent bed. If cartridge goes dry, repeat cartridge conditioning steps.

5.2 Handling of Extract

5.2.1 Before the silica gel cleanup technique can be utilized, the extract must be exchanged to hexane and the phenols must have undergone derivatization by pentafluorabenzyl bromide, as per the method.

5.2.2 Use the 2g conditioned silica gel cartridges.

5.2.3 Rinse the cartridge by using two 5 ml aliquots of hexane. Discard aliquots.

5.2.4 Transfer the extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 ml/minute.

5.2.5 When the entire extract has passed through to cartridges, but before the cartridge becomes dry, rinse the sample vials with an additional 5.0 ml of solvent. Add the rinse to the sample collection vial.

5.2.6 Close the cartridge valve and turn off vacuum after the solvent has passed through, ensuring the cartridge never goes dry. Replace the collection vial.

5.2.7 Add 5 ml of toluene/hexane (25/75, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial.

5.2.8 Adjust the final volume of the eluant to a known volume which will result in analyte concentrations appropriate for the project requirements using techniques described in an appropriate 3500 series method. The cleaned extracts are ready for analysis.

6.0 QUALITY CONTROL

6.1 Before any sample can be processed using silica cartridges, the analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentration. The efficiency of each lot of the solid-phase extraction cartridges must be verified. The recovery for each analyte must be greater than 85%. Only lots of cartridges from which the spiked analytes are quantitatively recovered are to be used to process the samples. A check is also performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

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Table 4
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (25ng DFTPP)	Every 12 hours.	Ensure correct mass assignment. DFTPP % Relative abundance criteria as specified in Table 3. Pentachlorophenol tailing < 2, Benzidine tailng < 2 DDT breakdown < 20%.	Retune. Do not proceed with analysis until DFTPP spectrum meets criteria.
Initial Calibration	Each time the instrument is set up and when CCCs and SPCCs in the continuing calibration verification (CCV) do not meet criteria. Established initially at minimum five concentration levels (six concentration levels if a second order {quadratic} curve is used) - low standard at or below project required reporting limit (PRRL), near but above method detection limits (MDL).	1. Average relative response factors (RRFs) for SPCCs ≥0.05 2. % RSD for RRFs for each CCC ≤30%. 3. % RSD for RRFs for all target compounds ≤15%. IF RF % RSD >15% use linear curve, r >=.99, or quadratic curve, r2 > = .990. (NELAC, QSM, or other programs/agencies require different criteria than stated here. Program and/or project specific criteria are to be followed as stated in their documents.)	Correct system and recalibrate. Criteria must be met before sample analysis can begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	At or near the mid-point of calibration range for all target compounds, CCCs, SPCCs and surrogates and prepared from second source standards.	1. RRF for SPCCs ≥0.05 2. % Deviation. for RRFs of each CCC <20%. 3 Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to four target compounds <= 40% RSD. 4 QSM, NELAC, or other programs/agencies require different criteria than stated here. Program and/or project specific criteria are followed as stated in their documents (ex: <25%D for QSM projects).	Correct system and recalibrate. Criteria must be met before sample analysis can begin. IF % relative standard deviation (RSD) >20%, then system must be inspected and problem corrected before sample analysis. If >20% RSD then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. Sample results reported that have %RSD failures must be qualified (Z). QSM allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification standards(CCV)	Every 12 hours. At or near the mid-point of calibration range for all target compounds, CCCs, SPCCs and surrogates and is prepared from standards used for calibration.	1. RRF for SPCCs ≥0.05. 2. % Deviation for RRFs of each CCC <20%. 3. Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to four target compounds = 40% RSD. 4. QSM, NELAC, or other programs/agencies require different criteria than stated here. Program and/or project specific criteria are followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis can begin. IF % RSD >20%, then system must be inspected and problem corrected before sample analyses. If >20% RSD correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. Sample results reported that have %D failures must be qualified (Z). QSM allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in CCV level of ICAL. 2. Retention time (RT) within 30 sec of RT for associated CCV standard. 3. QSM, NELAC, or other programs/agencies require different criteria than stated here. Program and/or project specific criteria are followed as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Reextract and reanalyze sample. * If reanalysis is outside limits the data is qualified (S). Follow specified criteria as stated in Shell or other documentation.
Method Blank (MB)	One per prep batch/20 samples per matrix. The MB is used to document contamination resulting in the analytical process and is carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern are to be less than the highest of either: Method detection limit, five percent of the regulatory limit for that analyte, or 1/5 of the measured concentration in the sample. 2. QSM: <1/2 MRL. Common lab contaminants < MRL.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples. For QSM data, if <1/2 MRL no action required. If no sample remains for re-prepping, or if re-prepped data still contains contamination, flag data with (B) qualifier.

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per prep batch of 20 samples. Must undergo all sample preparation procedures. Spiking solution are to contain all target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries (and RPDs, if applicable) within in-house generated limits. Default 70-130% (20% RPD). Sporadic marginal failure (SMF) of up to four target compounds = 60-140 % rec. 2. Use QSM, Project Specific or Client limits when applicable.	If LCS recoveries are within control limits or within SMF frequency and limits then no action is required. If the LCS exceeds control limits, as well as SMF criteria, reanalyze the LCS. If LCS recoveries are still outside control limits, reextract and reanalyze samples. If sample is not available for re-extraction then qualify data for the failing analytes with a (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.
MDL Level Verification MDL check (2X MDL)	Once per quarter per instrument	1. Analytes detected	Correct problem and repeat.
MRL Level Verification Check standard at Reporting Limit.	Beginning and End of 12 hr. sequence.	1. 70-130% or project specific/client limits 2. SMF 60-140%.	Run MDL check to verify that compounds can be detected.
Matrix Spike/Matrix Spike Duplicate	One set per prep batch of 20 samples. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries and RPDs within in-house generated limits. Default 70-130 / 20%RPD. 2. Use QSM, Project Specific or Client limits when applicable.	If LCS is acceptable, then report probable matrix interference. Qualify data if the recoveries are low (M). If recoveries are high and there are no detects in the unspiked sample then that data does not require flagging. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.

Table 4 (Continued)
Semivolatile Organic Compounds - Method 8270 Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Qualitative/Quantitative Issues	If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed.	The instrument level of all compounds must be within the calibration range for all samples.	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results are to be flagged (X).
Surrogate	1. Calibrated as target compounds. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. All % Recoveries within in-house generated limits. 2. Use QSM, Project Specific or Client limits when applicable.	If more than one recovery is not within limits: Check to be sure that there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance. If no problem is found, reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample is qualified (S).
Retention Time Window (RTW)	The position will be set using the midpoint of the calibration curve or the value in the CCV run at the beginning of the analytical sequence.	RTs of analytes must be within +/-.06 RRT units of the RRT of the CCV.	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset RTW.

Table 5
8270 Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MRL					
b. SPCC responses.	Avg. RRF ≥ 0.05					
c. Linearity.	RSD≤ 15%, ≤ 30% for CCCs, and r ≥ 0.990 for regression.					
d. Were the standards used for the ICAL uniquely identified?						
e. Was there a DFTPP standard analyzed prior to the ICAL?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the SPCC within QC limits	RRF ≥ 0.05					
c. Were the CCCs within QC limits	%D ≤ 20%					
d. Were the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs for target analytes analyzed at the beginning of the sequence and after every 12 hours.						
b. Were SPCC compounds acceptable?	RRF ≥ 0.050					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
c. Were the CCCs compounds acceptable?	%D ≤ 20%					
d. Were the recoveries for the CCVs acceptable?	%D≤20%, 4 analytes ≤40%.					
e. Was each CCV uniquely identified (i.e. Standard Number)?						
4. DFTPP						
a. Was a DFTPP tune check ran at the beginning of every twelve hour shift?						
b. Were the relative abundance criteria met?						
c. Was the peak tailing < 5 on Pentachlorophenol and < 3 on Benzidine?						
d. Was the breakdown of DDT to DDE and DDD less than 20%						
5. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Were the MB results less than the reporting limit (RL)?	< MRL					
If no, were positive hits in the samples < 5x the amount in the blank flagged with a "B".	< 5x (qualify data) > 5x (no action)					
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch < 20 samples					
6. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits					
If no, and the recoveries were low, flag those analytes "Q". If the recoveries were high, only flag the detects (>RL) for those analytes "Q".						
7. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits					
c. Is the relative percent difference (RPD) for each analyte between a matrix spike (MS) and matrix spike duplicate (MSD) within the acceptance criteria? (same criteria for LCS and LCSD)	20% RSD or In-house limits					
9. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. acid treatment, acid-base fractioning, and Florisil treatment)	GPC, Acid/Base, Florisil, Treatments					
10. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the data file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

CT Laboratories
Baraboo Laboratory Divison

Title: **TCLP and SPLP Extraction for Non-Volatile Parameters**

SOP Number: **CL-8B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 The following procedure will be used for performing the metals and semi-volatile extraction.

2.0 METHOD SUMMARY

2.1 TCLP

2.1.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.1.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.1.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

2.2 SPLP

2.2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are

analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 PB or MB (Prep Blank/ Method Blank) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch.

3.3 MS (Matrix Spike): - A separate sample aliquot to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. One MS is prepared for each waste type in a given batch of samples. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the sample.

4.0 HEALTH AND SAFETY

4.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

There are no cautions

6.0 INTERFERENCES

Refer to the sample analytical methods for interferences.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis should be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 Personnel should know how to read a meniscus and how to use a balance correctly.

8.0 APPARATUS AND MATERIALS

8.1 Rotator apparatus capable of turning at 30 +/- 2 rpm.

8.2 2 liter glass extraction jars.

8.3 2 liter polyethylene extraction containers.

8.4 Millipore pressure filtration apparatus. Note: The interior surface of the pressure filtration apparatus should be free smooth and free of scratches. Clean using only a very soft bristled brush if necessary. Also, the screen

on which the filter is placed should be clean of debris. If any of the holes are clogged they can be cleaned by sonicating for 15 minutes.

8.5 Glass fiber filters 0.7 micron: Environmental Express TCLP filters or equivalent.

8.6 Ceramic filtration funnel, 15 cm.

8.7 2 liter filtration flask.

8.8 pH meter.

8.9 Top-loading balance, 0.01 g capacity.

8.10 Hotplate.

8.11 Magnetic stirrer.

8.12 100 mL graduated cylinder.

8.13 5 mL oxford pipette.

8.14 Thermometer, 100^0C.

8.15 Reagents:

8.15.1 De-ionized water: Milli-Q type II

8.15.2 Hydrochloric acid 1N: Prepare in hood. Into a 1 liter volumetric flask, add 900 mL of D.I. H2O. Carefully add 83 mL ACS reagent grade conc. HCl. Dilute to volume with D.I. H2O and mix well.

8.15.3 Glacial acetic acid, conc. ACS reagent grade.

8.15.4 Sodium Hydroxide 10N: Into a 1 liter volumetric flask, add 500 mL of D.I. H2O. Dissolve 400g. ACS reagent grade NaOH pellets (caution: mixture will become very hot). When cool, dilute to volume with D.I. H2O and mix well.

8.15.5 SPLP extraction fluid acid mixture: To a 100 mL volumetric flask, add 50 mL D.I. H2O. Carefully add 6 g. Conc. H2SO4 and 4 g. Conc. HNO3. Mix by swirling and dilute to volume with D.I. H2O.

8.16 EXTRACTION FLIUDS

8.16.1 TCLP extraction fluid #1: (To prepare a 20 liter quantity): Fill a 20 L carboy with 19 L Of D.I. H_2O. Add 114 mL glacial acetic acid and 128.6 mL 10N NaOH. Dilute to 20 L With D.I. H_2O and mix by stirring. When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

8.16.2 TCLP extraction fluid #2: (To prepare a 20 liter quantity): Fill a 20 L Carboy with 19 L of D.I. H_2O. Add 114 mL glacial acetic acid. Dilute to 20 L and mix by stirring. When correctly prepared, the pH of this fluid will be 2.88 +/- .05.

8.16.3 SPLP extraction fluid #1: To be used for sites that are east of the Mississippi River. Prepare a sufficient quantity of extraction fluid by adding the SPLP acid mixture (see step 8.15.5 of this SOP) to D.I. H_2O to obtain a pH of 4.20 +/- 0.05. Note: solutions are unbuffered and the exact pH may not be obtained.

8.16.4 SPLP extraction fluid #2: To be used for sites that are west of the Mississippi River. Prepare a sufficient quantity of extraction fluid by adding the SPLP acid mixture (see step 8.15.5 of this SOP) to

D.I. H_2O to obtain a pH of 5.00 +/- 0.05. Note: solutions are unbuffered and the exact pH may not be obtained.

9.0 INSTRUMENT OR METHOD CALIBRATION

No calibration is necessary.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 Preservatives shall not be added to samples before extraction. Extracts to be analyzed for metals shall be preserved following filtration with conc. HNO_3. Extracts to be analyzed for Semivolatile organic compounds (SVOCs) shall not be preserved following filtration. Extracts to be analyzed for Phenolics shall be preserved following filtration with conc. sulfuric acid.

10.2 Sample hold times are as follows (days):

	SVOCs	Mercury	Metals	Phenolics
From sample date to TCLP extraction:	14	28	180	28
From TCLP extraction to preparative extraction:	7	n/a	n/a	n/a
From preparative extraction to analysis:	40	28	180	28

See TCLP method 1311 sec. 6.0 and SPLP method 1312 sec. 6.0 for a detailed description of sample handling.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 See appropriate SOP for sample analysis following extraction with this mehod.

11.2 DETERMINE SAMPLE % SOLIDS

11.2.1 For solid samples which contain no free liquids, proceed to sec. 11.3.2.

11.2.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required as follows:

11.2.2.1 Preweigh a GFF filter and record the weight.

11.2.2.2 Preweigh a receiving beaker and record the weight.

11.2.2.3 Preweigh a transfer beaker and record the weight.

11.2.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker beneath the outlet.

11.2.2.5 Weigh out a subsample of the waste (100g. minimum) and record the weight. An additional minimum 100g. will be needed for the extraction.

11.2.2.6 Transfer the waste to the filtration device and secure the top.

11.2.2.7 Re-weigh the empty transfer beaker and record the weight.

11.2.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through

the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.
*Note: Some wastes, such as oily wastes and some paint wastes will contain material that appears to be a liquid. Even after applying pressure to 50 psi, this material may not filter. In this case, the material in the filter holder is defined as the solid phase and is carried through the extraction as a solid. Proceed to sec. 11.4.

11.2.2.9 Weigh the receiving beaker and record the weight.

11.2.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.
*Note: This subsample is not to be used for the extraction procedure.

11.2.2.11 Determine and record the weight of the liquid phase.

11.2.2.12 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the waste.

11.2.2.13 Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of solid phase}}{\text{total weight of waste}} \times 100$$

11.3 EVALUATION OF % SOLIDS

11.3.1 If the % solids is <0.5%, the filtrate is defined as the TCLP extract. Proceed to section 11.5 to prepare the extract for analysis.

11.3.2 If the % solids is significantly >0.5%, the solid portion must have a particle size which is smaller than 1cm. or have a surface area >3.1 cm^2 /g. (paper, cloth, etc.).If the above is not met, the material must be reduced to particles of the appropriate size by cutting, crushing, or grinding.

11.3.3 If the % solids is $\geq$0.5% or is very close, and it is noticed that the solid material is entrained in the filter, dry the filter at 80-120C until two successive weighings agree within +/- 1%. Determine the % dry solids. If the % dry solids is <0.5%, the filtrate is defined as the TCLP extract. Proceed to section 11.5 . If the % dry solids is >0.5%, see note below.
*Note: there must be a significant level of % solids such that a minimum of 25-50g of solids can be generated for the extraction. This minimum amount of solids will yield 500-1000 mL of extract.

11.4 DETERMINE THE APPROPRIATE EXTRACTION FLUID

11.4.1 TCLP

11.4.1.1 Transfer 5.0 g. of a representative subsample of the waste to a clean 250 mL beaker.

11.4.1.2 Add 96.5 mL D.I. H2O and cover with a

watchglass.

11.4.1.3 Stir vigorously for 5 min.

11.4.1.4 Measure and record the pH.

*Note: Accurate pH measurement is critical; calibrate the pH meter daily with fresh buffer solutions.

11.4.1.5 If the pH is <5.0, use extraction fluid #1.

11.4.1.6 If the pH is >5.0, add 3.5 mL of 1N HCl, cover with a watchglass, and heat on a hotplate to 50^0 C. Hold at 50^0 C for 10 min. Cool to room temperature, measure and record the pH. If the pH is < 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

11.4.2 SPLP

11.4.2.1 For samples collected from sites east of the Mississippi River, use extraction fluid #1.

11.4.2.2 For samples collected from sites west of the Mississippi River, use extraction fluid #2.

11.5 EXTRACTION PREPARATION

11.5.1 For samples that are 100% total solids

11.5.1.1 Perform particle size reduction if necessary.

11.5.1.2 Weigh **at least** 100 g of sample directly into the extraction vessel and record the weight. (For SVOC and phenolics analysis, a glass container must be used. For metals analysis, a glass or plastic container may be used.)

11.5.1.3 Record the number of the vessel being used and also check it off on the vessel usage sheet in front of the TCLP extraction log. A blank must be run on each container at a minimum of every 20th use.

11.5.1.4 Determine the amount of extraction fluid to add to the extraction container as follows:
Volume of extraction Fluid (mL) = 20 x mass of sample (g)

11.5.1.5 Measure and record the pH of the extraction fluid immediately prior to use.

11.5.1.6 Add the appropriate amount of extraction fluid.

11.5.1.7 Measure and record the initial pH.

11.5.1.8 Tightly cap the extraction container. The sample is now ready for extraction. Proceed to sec. 11.6.

11.5.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required on a new portion of the waste:

11.5.2.1 Preweigh a GFF filter and record the weight. Filters should be prewashed with 1N HNO_3 followed by D.I. H_2O if metals are to be analyzed.

11.5.2.2 Preweigh a receiving beaker and record the weight.

11.5.2.3 Preweigh a transfer beaker and record the weight.

11.5.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker underneath.

11.5.2.5 Weigh out at least 100 g of waste and record the weight. Ideally, enough sample should be filtered to allow for 100 g of solids to remain.

11.5.2.6 Transfer the waste to the filtration device and secure the top.

11.5.2.7 Re-weigh the empty transfer beaker and record the weight.

11.5.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

11.5.2.9 Weigh the receiving beaker and record the weight.

11.5.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

11.5.2.11 Measure and record the weight and volume of the liquid phase.

11.5.2.12 Using a transfer pipette and a small beaker, add a few drops of the liquid phase to a small quantity of D.I. H_2O.

11.5.2.13 If the two phases are miscible, save the liquid phase for addition back to the filtered TCLP or SPLP extract. Store at 4^0 C in an appropriate container until the extraction is complete. If the two phases do not mix, the liquid phase will need to be analyzed separately and the results mathematically combined with the results from the extract.

Calculation:

Final analyte concentration = $$\frac{(V1)(C1) + (V2)(C2)}{V1 + V2}$$

V1 = volume of the first phase liquid
C1 = concentration of the first phase in mg/L
V2 = volume of the second phase liquid
C2 = concentration of the second phase in mg/L

11.5.2.14 Disassemble the filtration apparatus and carefully remove the filter and waste. Perform particle size reduction if necessary. Quantitatively transfer the filter and waste into the appropriate extraction container.

11.5.2.15 Determine the amount of extraction fluid to add to the extraction container as follows:

Volume of extraction Fluid (mL) = 20 x mass of sample (g)

11.5.2.16 Add the appropriate amount of extraction fluid.

11.5.2.17 Measure and record the initial pH.

11.5.2.18 Tightly cap the extraction container. The sample is now ready for extraction.

11.6 EXTRACTION

11.6.1 Secure the samples in the rotation apparatus which is located in the BOD incubator

11.6.2 Rotate for 18 +/- 2 hours. A room temperature of 23 +/- 2^0C must be maintained during the extraction period. This can be checked in the electronic temperature data logger.

11.6.3 Begin rotating. Record the time.

11.7 FILTRATION FOLLOWING EXTRACTION

11.7.1 Record the time and temperature at the end of the extraction period, as well as the minimum and maximum temperatures.

11.7.2 Following the rotation period, measure and record the pH of each bottle

11.7.3 Assemble the 2 L vacuum flask and ceramic filtration funnel.

11.7.4 Obtain the necessary sample containers and pH paper.

11.7.5 Place a 0.7 micron glass fiber filter (Environmental Express TCLP filters) in the filtration funnel. Record the lot # of filter that is used.

11.7.6 Filter the extract.

11.7.7 Transfer a suitable quantity of the filtrate to the appropriate sample containers:

Metals: 250 mL in polyethylene with HNO_3 preservative.
Semi-volatiles and phenolics: 1 L in amber glass jar.

11.7.8 For metals analysis, preserve the sample to pH <2 with conc. HNO_3.

11.7.9 For semi-volatile analysis, preservative is not added. Store at 4 ^{o}C.

11.7.10 For phenolics analysis, preserve the sample to pH<2 with conc. H_2SO_4.

12.0 TROUBLESHOOTING AND MAINTENANCE

12.1 There is no troubleshooting or maintenance for this method.

13.0 DATA ACQUISITION, CALCULATION AND REDUCTION

13.1 See section 11.0 for any applicable calcuations.

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Data is recorded in the TCLP/SPLP extraction log.

15.2 Prep data is entered into LIMS, and then the batch sheet is given to the metals prep analyst or the semi-volatile prep analyst.

16.0 QUALITY CONTROL/QUALITY ASSURANCE

16.1 A minimum of one blank, using the same extraction fluid as used for the samples, is required for every extraction batch. Also, a blank must be performed every 20th time an extractions is performed in a particular container to check for contamination.

16.2 A matrix spike is required for each waste type, with a minimum of one matrix spike per extraction batch. Matrix spikes are prepared at the time of digestion/analysis.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1311.

17.2 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1312.

CT Laboratories
Baraboo Laboratory Divison

Title: TCLP and SPLP Extraction for Volatile Organic Compounds

SOP Number: CL-8A

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance *Date*

_____
Laboratory Director *Date*

SOP Manual Control Number:_____

1.0 SCOPE AND APPLICATION

1.1 The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.The following procedure will be used for performing the volatile organic ZHE procedure.

2.0 METHOD SUMMARY

2.1 TCLP

2.1.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.1.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.1.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

2.2 SPLP

2.2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2.2 For samples containing greater than 0.5 % solids, the liquid phase,if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 PB or MB (Prep Blank/ Method Blank) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch.

3.3 MS (Matrix Spike): - A separate sample aliquot to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. One MS is prepared for each waste type in a given batch of samples. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the sample.

4.0 HEALTH AND SAFETY

4.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

5.1 For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced.

5.2 The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device.

6.0 INTERFERENCES

6.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis should be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 Personnel should know how to read a meniscus and how to use a balance correctly.

8.0 APPARATUS AND MATERIALS

8.1 Rotator apparatus capable of turning at 30 +/- 2 rpm.
8.2 ZHE extraction devices.
8.3 Millipore pressure filtration apparatus.
8.4 Glass fiber filters 0.6-0.8 micron 11.0 cm. (Whatman GFF or equivalent).
8.5 Glass fiber filters 0.6-0.8 micron: 15.0 cm. (Whatman GFF or equivalent).
8.6 Fluid pump.
8.7 Gas pressure/vacuum pump.
8.8 250 mL and 500 mL beakers.
8.9 100mL glass gas tight syringe.
8.10 Tedlar bags.
8.11 40 mL VOA vials.
8.12 Top-loading balance.
8.13 TCLP/SPLP prep log.
8.14 Reagents
8.14.1 De-ionized water: Milli-Q type II
8.14.2 Extraction fluids:
8.14.2.1 **TCLP extraction fluid 'A'**: (To prepare a 20 liter quantity):
Fill a 20 L. Carboy with 19 L. Of D.I. H2O. Add 114 mL 1N glacial acetic acid and 128.6 mL 10N NaOH. Dilute to 20L. With D.I. H2O and mix by stirring. Adjust the pH with acetic acid or NaOH to obtain 4.93 +/- 0.05.
8.14.2.2 **SPLP extraction fluid**: reagent water

9.0 INSTRUMENT OR METHOD CALIBRATION

There is no calibration for this method.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 Preservatives shall not be added to samples before extraction.
10.2 Care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g.,samples should be collected in Teflon-lined septum capped vials and stored at 4C. Samples should be opened only immediately prior to extraction). Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.
10.3 Volatiles have 14 days from the date of collection to be extracted.
10.4 Volatiles have 14 days from the date of extraction to be analyzed.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 **Determine sample % solids:**
11.1.1 For solid samples which contain no free liquids, proceed to sec. 11.3.

11.1.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required as follows:

11.1.2.1 Preweigh a GFF filter and record the weight.

11.1.2.2 Preweigh a receiving beaker and record the weight.

11.1.2.3 Preweigh a transfer beaker and record the weight.

11.1.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker underneath.

11.1.2.5 Weigh out a subsample of the waste (100g. minimum) and record the weight. An additional minimum 25g. will be needed for the extraction. For low volume samples, consult your supervisor.

11.1.2.6 Transfer the waste to the filtration device and secure the top.

11.1.2.7 Re-weigh the empty transfer beaker and record the weight.

11.1.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

*Note: Some wastes, such as oily wastes and some paint wastes will contain material that appears to be a liquid. Even after applying pressure to 50 psi, this material may not filter. In this case, for the non-volatile extraction, the material in the filter holder is defined as the solid phase and is carried through the extraction procedure as a solid.However, since the volatile procedure requires utilizing the ZHE device to filter the sample following rotation, the ZHE filter may become plugged and filtration of the extract from the ZHE device may not be possible.

11.1.2.9Weigh the receiving beaker and record the weight.

11.1.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

11.1.2.11 Determine and record the weight of the liquid phase.

11.1.2.12. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the waste.

11.1.2.13. Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of solid phase}}{\text{total weight of waste}} \times 100$$

11.2 Evaluation of % solids:

11.2.1 If the % solids are <0.5%, a fresh portion of the waste will be filtered through the ZHE device and collected in a Tedlar bag or a 100 mL syringe. This filtrate will be defined as the TCLP extract. Proceed to sec.11.3, then 11.4.4

11.2.2 If the % solids are significantly >0.5%, proceed to section 11.3, then 11.4.1. or 11.4.2.

11.2.3 If the % solids are ≥0.5% or are very close, and it is noticed that the solid material is entrained in the filter, dry the filter at 80-120C until two successive weighings agree within +/- 1%. Determine the % dry solids. If the % dry solids are <0.5%, follow 11.2.1. If the % dry solids are >0.5%, see note below.
*Note: there must be a significant level of % solids such that a minimum of 5-10g of solids can be generated for the extraction. This minimum amount of solids will yield 100-200 mL of extract.

11.3 ZHE device preparation:

11.3.1 Assemble the ZHE device as follows:

11.3.1.1 Place two O-rings on the piston.

11.3.1.2 Place an O-ring in the ZHE base.

11.3.1.3 Wet the O-rings of the piston and place the piston inside the ZHE body. Depress the piston into the ZHE body only far enough to allow room for the sample.

11.3.1.4 With the piston installed, place the ZHE body into the base.

11.3.1.5 Place an O-ring on the top of the ZHE body.

11.3.1.6 Place a 11.0 cm. GFF filter between the two filter screens and set aside.

11.3.1.7 The ZHE device is now ready to receive a sample.

11.4 Adding sample to the ZHE device:

11.4.1 For samples that are 100% total solids:

11.4.1.1 Weigh out 25.0g. of sample into a beaker or other suitable container and record the weight. If particle size reduction if required, chill the sample and reduction equipment to 4^0C to reduce the loss of volatile compounds. Proceed to chop, crush, or grind the sample to a minimum $1cm^2$ size. Minimize exposure to the atmosphere.

11.4.1.2 Transfer the waste material to the ZHE device and secure the top.

11.4.1.3 Attach the air line from the pressure pump to the lower valve. Open the upper and lower valves.

11.4.1.4 Slowly pressurize the ZHE device to 50 psi to force the piston to the top of the ZHE body, thereby removing any headspace. Remove the air line.

11.4.1.5 Determine the amount of extraction fluid to add follows: amount of fluid added = 20 x's the weight of the sample

11.4.1.6 Proceed to sec. 11.5

11.4.2 For samples >5% but <100% total solids:

11.4.2.1 Determine the amount of sample to add to the ZHE as follows:

Weight of sample to use = $\frac{25}{\quad}$ x 100

% total solids (sec. 11.1)

11.4.2.2 Proceed to sec. 11.4.3, using the amount of sample determined above.

11.4.3 For samples that are between 0.5% - 5% total solids:

11.4.3.1 Weigh a 500 mL beaker and record the weight.

11.4.3.2 Into the beaker, weigh out 500 g. of sample and record the weight.

11.4.3.3 Transfer the waste to the ZHE device and secure the top.

11.4.3.4 Connect the air line to the lower valve. Open upper and lower valves.

11.4.3.5 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

11.4.3.6 Pre-weigh a Tedlar bag and record the weight.

11.4.3.7 Slowly pressurize the ZHE to expel the liquid into the bag. Do not exceed 50 psi.

11.4.3.8 Close the valves. Remove the bag, weigh the bag and it's contents, and record the weight.

11.4.3.9 Determine the amount of extraction fluid to add follows:

$$\text{weight of extraction fluid} = \frac{20 \times \text{weight of sample waste} \times \%\ \text{solids}}{100}$$

11.4.3.10 Proceed to sec. 11.5

11.4.4 For samples that are <0.5% total solids:

11.4.4.1 The liquid portion of the waste, after filtration is defined as the TCLP extract. The ZHE device will be used to filter the sample.

11.4.4.2 Add an appropriate amount of sample to the ZHE device to complete the requested analyses.

11.4.4.3 Secure the top, connect the air line to the lower valve. Open upper and lower valves.

11.4.4.4 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

11.4.4.5 Connect a 100 mL syringe or evacuated Tedlar bag to the top valve. Slowly pressurize the ZHE to force the liquid into the syringe or bag. If collecting the liquid with a syringe, carefully transfer, (after discarding the first 5 mL) with headspace, to 40 mL VOA vials. If using a bag, allow the liquid to flow into the bag until a sufficient quantity has been collected for analysis. Store the extract at 4^0C until analysis.

11.5 Adding extraction fluid to the ZHE device:

11.5.1 Transfer the appropriate amount and type of extraction fluid to a 500 mL beaker.

11.5.2 Using the fluid pump, place the intake line into the beaker of extraction fluid. Turn on the pump, and allow the fluid to enter the

pump. Stop the pump when the fluid appears at the end of the outlet line.

11.5.3 Connect the outlet line to the ZHE top valve. Turn on the pump, and allow the pump to charge the ZHE with the entire contents of the beaker. Stop the pump before any air reaches the ZHE. Close the top and bottom valves, and remove the inlet line. Manually rotate the ZHE device in an end over end fashion 2 or 3 times. *Note: while the ZHE is filling, check for fluid leaking out of the bottom valve. If this happens, stop the pump and use a different ZHE device with a fresh sample.

11.5.4 Attach the air line to the bottom valve. Open the top valve, and while holding a paper towel at the valve, slowly pressurize the ZHE to expel any remaining air. Close the valve at the first sign of fluid at the valve.

11.5.5 With the air line still attached, re-pressurize the ZHE device to 10 psi. Check for leaks. The ZHE is now ready for the 18 +/- 2 hr. rotation. Proceed to sec. 11.6.

11.6 Extraction:

11.6.1 Secure the samples in the rotation apparatus.

11.6.2 Rotation time is 18 +/- 2 hours at 30 +/- 2 rpm. A room temperature of 23 +/- 2^0C must be maintained during the extraction period. A min/max thermometer shall be used to monitor the temperature.

11.6.3 Begin rotating. Record the time and room temperature.

11.6.4 Following the rotation period, record the end time of rotation. Proceed to sec 11.7 for filtering the extract.

11.7 Filtration following extraction:

11.7.1 Following the rotation period, check the ZHE pressure gauge to ensure that the device did not leak. If pressure was not maintained, the sample must be re-extracted with a new portion of waste.

11.7.2 If pressure has been maintained, the material in the ZHE device is separated into liquid and solid phases.

11.7.3 For samples with no initial liquid phase**:**

11.7.3.1 Attach the 100 mL gastight syringe to the ZHE outlet valve. Open the valve and carefully withdraw the extract into the syringe. Do not allow air bubbles into the syringe. Pressurizing the ZHE to 10 psi may help in withdrawing the extract.

11.7.3.2 Discard the first 5 mL out of the syringe, then transfer the remaining extract to 40 mL VOA vials (without headspace). Store at 4^0C until analysis.

11.7.4 For samples containing an initial liquid phase**:**

11.7.4.1 If an initial liquid phase was collected, determine if the ZHE liquid extract will mix with the initial liquid phase:

11.7.4.1.1 Using a transfer pipette and a small beaker, add a few drops of the liquid phase to a small quantity of D.I. water. Observe to see if the two liquids are miscible, or if they separate into layers.

11.7.4.1.2 If the two phases are miscible, combine the filtered extract with the initial liquid phase and analyze as one sample. Attach the Tedlar bag containing the initial liquid phase to the outlet valve of the ZHE device. Gradually apply pressure to the lower valve and slowly filter all of the extract into the Tedlar bag. Store at 4^0C until analysis.

11.7.4.1.3 If the two phases do not mix, the initial liquid phase and the filtered extract will need to be collected and analyzed separately. Collect all of the extract in a separate Tedlar bag.

11.7.4.2 Calculate the results as follows:

$$\text{Final analyte concentration} = \frac{(V1)(C1) + (V2)(C2)}{V1 + V2}$$

V1 = volume of the first phase liquid
C1 = concentration of the first phase in mg/L
V2 = volume of the second phase liquid
C2 = concentration of the second phase in mg/L

12.0 INSTRUMENT MAINTENANCE AND TROUBLESHOOTING

13.0 DATA ACQUISITION, CALCULATION AND REDUCTION

13.1 Refer to section 11 for necessary calculations

14.0 COMPUTER HARDWARE AND SOFTWARE

14.1 Computer with StarLIMS

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Data is recorded in the TCLP/SPLP extraction log.

15.2 Prep data is entered into LIMS, and then the batch sheet is given to the metals prep analyst or the semi-volatile prep analyst.

16.0 QUALITY CONTROL/QUALITY ASSURANCE

16.1 A minimum of one blank, using the same extraction fluid as used for the samples, is required for every extraction batch. Also, a blank must be performed every 20th time an extractions is performed in a particular container to check for contamination.

16.2 A matrix spike is required for each waste type, with a minimum of one matrix spike per extraction batch. Matrix spikes are prepared at the time of digestion/analysis.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1311.

17.2 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1312.

CT Laboratories .
Baraboo Laboratory Division

Title: ***Analysis of Volatile Organic Compounds by GC/MS***

SOP Number: **5280B**

Prepared by: _____
Date

Technical Review by: _____
Date

Reviewed by: _____
Quality Assurance ***Date***

_____
Laboratory Director ***Date***

SOP Manual Control Number:

1.0 Identification of Test Method

1.1 This method is designed to follow procedures and QC requirements found in EPA SW-846 methods 5030, 5035, 8000 and 8260 in order to determine quantities of volatile organic compounds found in a variety of different sample matrices.

2.0 Applicable Matrix or Matrices

2.1 Volatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, soils and sediments, as well as other matrices noted in SW-846 method 8260B.

3.0 Detection Limits

1.1 Method detection limits (MDLs) are determined annually and results vary from compound to compound. Water MDLs typically fall in the range of 0.10 to 10 ug/L (5.0 ml purge) or 0.02 to 2.0 ug/L (25 ml purge). Soil MDLs are usually found to be between 0.0001 to 0.005 mg/kg (low-level soils) or 0.005 to 0.50 mg/kg (methanol preserved). Procedures for conducting MDL studies can be found in CT Laboratories Initial Method Performance and Reporting SOP.

4.0 Scope and Application

4.1 This method is used to quantify Volatile Organic Compounds (VOCs) with boiling points below 200° Celsius (°C) in water and soils. See Table 1 for typical target analyte list (TAL).

4.2 Examples of other compounds which have been analyzed by this method include: iodomethane, 2,3-dichloro-1-propene, 1-chlorohexane, acrolein, acrylonitrile, 1,1,2-trichloro-1,2,2-trifluoroethane, ethyl ether, hexane, ethyl acetate, 1-chlorohexane, and 2-Chloroethylvinyl ether. SW-846, method 8260B notes a number of other compounds amenable to this test.

5.0 Method Summary

5.1 A Purge & Trap system (including autosampler), a Gas Chromatograph (GC), and a Mass Spectrometer (MS) are utilized for the detection of VOCs. The autosampler introduces the sample to the purge and trap concentrator. The concentrator then removes the volatile constituents by purging the sample with an inert gas (Helium). The constituents are then collected onto an adsorption trap. The trap is then rapidly heated and the volatilized compounds are introduced to the GC. The GC is temperature programmed to facilitate separation of the individual organic compounds. Finally the separated compounds enter the MS (which is interfaced with the GC) for quantitative and qualitative analyses.

5.2 Utilizing computer software, identification of target analytes is accomplished by comparing the mass spectra of the sample constituent with that of commercially purchased standards. Quantitation is achieved by comparing the response of a quantitation ion relative to an

internal standard using a five point (minimum) calibration curve.

6.0 Definitions

6.1 4-Bromofluorobenzene (BFB), is used to verify that the GC/MS is properly tuned and ready for calibration and sample analyses. BFB is directly injected at 25-50 ng and precedes all other analyses. A twelve hour analysis time window is used before another BFB injection is required.

6.2 An Initial Calibration Verification standard (ICV) is analyzed before the analysis of samples as a check on the calibration curve. The ICV shall be of a second source, meaning a separate vendor and/or lot # shall be used to prepare this standard. A second source is needed to validate the integrity and concentration of the standards used for calibration.

6.3 An Initial Calibration Blank (ICB) is also analyzed before samples to confirm that the deionized water (DI H2O) used for standards, blanks, and dilutions is free from contamination. An ICB for soil samples also includes organic free sand and/or methanol used for standard and QC preparation, as well as sample dilutions and volume adjustments; the control sand and methanol must also be documented as free from contamination.

6.4 A Calibration Check Verification standard (CCV) is analyzed at the beginning of each sequence (following the BFB injection) shift and every 12 hour shift thereafter. A CCV is made up from the same standards used for calibration and is used to ensure that the calibration and retention times are stable.

6.5 An analytical batch is defined as a group of samples (not to exceed 20 samples) analyzed per sequential run. Depending on client or program requirements, an analytical batch may or may not include matrix spikes as part of the 20 samples.

6.6 Internal standards (ISTD) are added to each sample, blank, spike, and check standard. The ISTDs used are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The ISTD is used for internal calibration as a reference peak and to check purging efficiency.

6.7 Surrogate standards (SSTD) are also added to each sample, blank, spike, and check standard. The SSTDs used are Toluene-d_4, 4-Bromofluorobenzene, 1,2-Dichloroethane-d_4, and Dibromofluoromethane. The SSTD is used as matrix interference and as a method control check.

6.8 A Method Blank (MBW = water, MBS = soil) is analyzed with every batch of samples to ensure the instrument is free from contamination. Method blanks are also used to demonstrate that the DI H2O, organic free sand, and methanol used in sample preparation is also free from contamination.

6.9 A Continuing Calibration Blank (CCB) is analyzed every 12 hour shift after the MB has been analyzed and before sample reanalyzes to ensure the system is still free from contamination.

6.10 Instrument blanks (IB) are analyzed whenever instrument contamination is a suspected possibility. The purpose of an IB is to prevent carry over from a standard, spike, or contaminated sample into another sample or blank. The acceptance criteria for an IB is the same as for a MB or CCB (for example: if an IB contains detections above acceptance

criteria and a MB and/or sample following the IB exhibit the same contamination above acceptance criteria, then the instrument shall be considered contaminated). The samples and/or MBs shall be reanalyzed after the instrument is confirmed free from contamination.

6.11 A Matrix Spike and Matrix Spike Duplicate (MS/MSD) are analyzed with every batch of samples as a matrix interference and control check..

6.12 A Laboratory Control Spike (LCS) is analyzed with every sample batch as a system control and method control check.

6.13 Program/project/contract specific: Samples may be analyzed using any given program or project specific criteria. These programs/projects include, but are not limited to, the WI-Underground Storage Tank (UST or LUST) program, the Louisville Chemistry Guidelines (LCG), the Department of Defense-Quality Systems Manual (DoD/QSM) and the National Environmental Laboratory Accreditation Program (NELAP).

7.0 Interferences

7.1 Volatile materials in the laboratory and impurities in the purging gas and sorbent trap can cause significant amounts of background contamination. Improper tubing such as certain plastics and rubber shall not be used. The analysis of IBs and MBs will indicate as to whether or not this type of contamination is present. Since subtraction on background contamination is not allowed, care must be taken to eliminate this type of contamination.

7.2 Carry over contamination is a problem when a highly contaminated sample is followed by a clean sample. Rinsing the autosampler and concentrator and adequate baking of the trap can greatly reduce contamination from carry over.

7.3 Some samples contain a lot of water soluble materials, suspended solids, compounds with high boiling points, or target analytes with very high concentrations which may contaminate some or all of the analytical system. Removing components of the system for cleaning or cleaning of the entire system may be required to eliminate the interferences.

7.4 Compounds with poor purging efficiencies may remain in the purge system, particularly with 25 ml purges. Ensuring adequate rinsing and increased line temperatures will help reduce this problem.

7.5 All chromatography gas/purge lines shall be stainless steel or copper to prevent permeation from possible background contaminants (i.e. Methylene chloride). Background levels of Methylene chloride are possible so care needs to be taken to reduce this possibility. Analyst clothing previously exposed to Methylene chloride must not be worn and isolating the instruments from possible air born contamination is essential in reducing Methylene chloride background contamination.

7.6 A trip blank normally accompanies sample in shipment and storage as a check on possible contamination from volatile organics by diffusion through the septum seal in sample vials/containers.

7.7 Mass spectrometer sensitivity, column degradation, and contamination can also contribute to background interferences. Proper maintenance procedures on instrumentation is essential to continually producing quality data. Maintenance manuals are provided with each piece of

equipment and are essential for proper instrument care. The manuals used are as follows: Autosampler (Archon Purge and Trap Autosampler System Operator' Manual, Varian Model 5100;4552), Concentrator (LCS 2000 User Manual Tekmar and Purge & Trap Concentrator, Tekmar 3000), Gas Chromatograph--HP 5890 Series II Operating Manual, and Mass Spectrometer--HP 5972A MSD Hardware Manual). The presence of semi-volatile hydrocarbons need also be taken into consideration, so appropriate post analysis bake out times need to be incorporated.

8.0 Safety

8.1 Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

9.0 Equipment and Supplies

9.1 40 ml screw cap "VOA" vials-borosilicate glass with a Teflon faced silicone septum (QEC or equivalent).

9.2 2 oz., 4 oz., or 60- ml Teflon lined screw top sample jars (QEC or equivalent).

9.3 5 g or 25 g samplers for low level soils (Encore)

9.4 Top loading balance sensitive to 0.01 g (Mettler-Toledo, BD202).

9.5 pH paper to confirm water sample preservation(Color pHast, EM Reagents)

9.6 Stainless steel spatulas

9.7 10, 25, 50, 100, 500, and 1000 uL gas tight syringes for sample dilutions and standard preparation (Hamilton or equivalent).

9.8 5.0, 10.0, 25.0, 50.0 ml syringes with luer-lok tips for methanol preserved soil sample preparation and sample dilutions (Hamilton/SGE or equivalents).

9.9 10, 50 100, 200, 1000, and 2000 ml Volumetric flask for sample dilutions and standard preparation (Class A, Pyrex/Kimble or equivalents).

9.10 Auto pipetter – 2.5 to 25.0 ml – for dispensing methanol (Dispensette).

9.11 Sonicator used for methanol-preserved soil sample extraction (Fisher, FS-28 or equivalent).

9.12 Auto sampler used for sample introduction to the Purge and Trap (EST-Archon or equivalents).

9.13 Purge and Trap concentrator (Tekmar, 2000/3000 or equivalents)

9.13.1 The glass purging tubes are of 5 ml or 25mL size. The all-glass purging device shall be designed to accept 5 or 25 ml samples with a water column at least 5 cm deep. The smaller (5 ml) purging device is recommended if the GC/MS system has adequate sensitivity to obtain the method detection limits required for a specific project or program.

9.13.2 The trap currently used is Supelco Type K. As required by SW-846 methods, the trap must be at least 25 cm long and have an inside diameter of at least 0.105 inches. Starting from the inlet, the trap contains 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: 33% of 2,6-diphenylene oxide polymer, 33% of silica gel, and 33% of coconut charcoal.

9.14 Gas Chromatograph/Mass Spectrometer Data Systems (GC/MS).

9.14.1 Hewlett Packard Gas Chromatographs (5890 & 6890).

9.14.1.1 Columns, Supelco (SPB-624) or Zebron (ZB-624), 30 Meter x 0.25 mm ID, 1.4 um film thickness.

9.14.2 Hewlett Packard 5972 & 5973 Mass Spectrometers.

9.14.3 Hewlett Packard Chemstation Data Management System (version G1701AA v. A.03.02 for the 5972's) and MSD Chemstation (version D.01.02.16.15 for the 5973) with Enviroquant and Prolab data processing software.

10.0 Reagents and Materials

10.1 Purge and trap grade methanol: (Fisher, Purge & Trap grade or equivalent), stored in laboratory warehouse.

10.2 Reagent grade water, organic free (Milipore, 18 mega ohm quality).

10.3 Certified Calibration Standards: (VOC Mix--2000 ug/ml, Accustandard--#M-502-10X; Additions--1000/10,000 ug/ml, Protocol--#XCTIVOA-Rev or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

10.3.1 A 100 ug/mL[1] Continuing Calibration Verification (CCV) working standard is prepared by adding 150 uL of the VOC mix and 300 uL of the Additions mix to 2550 uL of methanol in a 3 ml Mininert vial.

10.4 Certified Calibration Check Standards: (VOC Mix--2000 ug/ml, Ultra Scientific--#DWM-588; Additions--1000/10,000 ug/ml, Protocol--#XCTIVOA-Rev or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

10.4.1 A 100 ug/mL[1] Initial Calibration Verification (ICV) working standard is prepared by adding 150 uL of the VOC mix and 300 uL of the Additions mix to 2550 uL of methanol in a 3 ml Mininert vial.

10.5 Certified Internal Standards: (ISTD Mix--2500 ug/ml, Restek--#30241 or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

10.5.1 An 80 ug/ml ISTD working standard is prepared by adding 320 of the ISTD mix to a 10 ml volumetric flask and brought to volume with methanol.

10.6 Certified Surrogate Standards: (SSTD Mix, Restek--#30240; 1,2-DCA-d_4 Ultra Scientific--#STS210, or equivalents[2]), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

10.6.1 A 100 ug/ml SSTD working standard is prepared by adding 120 uL of the SSTD mix and 150 ul of 1,2-DCA-d_4 to 2730 ml of methanol in a 3 ml mininert vial.

10.6.2 An 80 ug/ml ISTD/SSTD working standard is prepared by adding 320 uL of the ISTD Mix (sec. 10.5), 320 uL of the SSTD Mix (sec. 10.6), and 400 uL of 1,2-DCA-d_4 (sec. 10.6) to a 10 ml volumetric flask and brought to volume with methanol.

10.7 Certified Tuning Standard: 4- bromofluorobenzene {BFB} (Ultra Scientific—2000 ug/ml, #STS-110N or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

10.7.1 A 50 ug/ml working standard is prepared by adding 75 uL of the Certified standard to 2925 ml of methanol in a 3 ml mininert vial.

10.8 Sodium bisulfate (JT Baker--#3534-01 or equivalent), stored in cabinet in Volatiles laboratory.

10.9 All certified stock standards use the expiration date provided by the manufacturer/supplier. The subsequent working standards expire one week after preparation. When standards used for calibration are prepared from freshly open stock standard vials, the expiration of working standards used from that point on can be extended. They can be extended if the integrity of those standards can be confirmed and documented. For example, if an ISTD/SSTD or a CCV/ICV standard continues to produce acceptable results after one week from preparation, it can be assumed still valid.

[1] Due to lower response or purging efficiencies, a number of compounds are purchased and prepared at concentrations greater than 100 ug/ml. Those compounds and concentrations are noted on the calibration curve.

[2] This compound is needed for Method 524.2 and is not used for this method.

11.0 Sample Preservation and Storage

11.1 Water samples are stored at 4° C (+/- 2°). The sample storage area must be free of organic solvent vapors and direct or intense light. Samples are stored in the Volatiles lab in a double door refrigerator (separate from analytical standards).

11.1.1 Analyze properly preserved samples (pH <2) samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected. If samples are not preserved then they must be noted (or qualified) as improperly preserved.

11.1.2 Samples analyzed for Acrolein and Acrylonitrile are to be preserved at a pH of 4-5 and analyzed within 14 days (3 days if unpreserved).

11.1.3 If reactive compounds such as 2-Chloroethyl vinyl ether are target compounds than no preservatives are added and the sample needs to be analyzed as soon as possible.

11.1.4 Samples containing residual chlorine require alternative preservation (ascorbic acid or sodium thiosulfate) to reduce the chlorine. These sample shall be reduced to a pH of <2 (using HCL or $NaHSO_4$), to meet the 14 day is the hold time.

11.2 Soil samples are stored at 4° C (+/- 2°). The sample storage area must be free of organic solvent vapors and direct or intense light Samples are stored in a double door refrigerator located in the laboratory warehouse.

11.2.1 Samples received for low level analysis in "Encore" samplers must be preserved within 48 hours from time of collection. To preserve a sample, weigh it into a 40 ml VOA vial, record the weight, then add 0.2 grams of sodium bisulfate per 1.0 gram of sample. Finally add 5.0 ml of DI H2O and a stir bar. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

11.2.2 Samples received for low level analysis in "Terra Core" sampler vials are already preserved with bisulfate at 0.1 g per 1.0 gram of sample. The preweighed vial weight (tare weight which includes the weight of vial + 5 ml of preservative & a stir bar) is subtracted from the total weight of the vial to determine sample weight. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

11.2.3 Samples received in filled 2 oz. or 4 oz. jars can be weighed and prepared for low level analysis as described in section 11.2.1. or they can be weighed into a VOA vial and preserved at a 1:1 ratio with methanol for medium/high- level analysis. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

11.2.4 Samples collected and preserved with methanol in the field in preweighed 60-ml jars are weighed as is. The preweighed jar weight, as well as the methanol weight (19.8 grams for 25 ml of methanol) is subtracted from the total weight of the jar to determine sample weight. For Wisconsin LUST samples if the weight to volume ratio is more than 1:1 then methanol is added using the auto pipetter to correct the ratio to 1:1. Unless instructed by the client to do otherwise, the maximum acceptable weight for volume correction is 35 grams. If samples are being analyzed for the Wisconsin LUST program then the hold time is 21 days from collection. Otherwise hold time is 14 days.

11.2.5 Samples collected and preserved in the field using "Terra Core" sample vials are weighed as is. The preweighed jar weight (tare weight which includes weight of the

vial + MeOH) is subtracted from the total weight of the vial to determine sample weight. Unless instructed to do so samples are not adjusted for volume to weight differences. Analyze sample within 14 days from collection.

11.2.6 All soil sample are weighed on the top loading balance which is connected to a computer so that all weights can be automatically entered onto an Excel spread sheet. The Excel spreadsheet is set up to record the weights as well as calculate the methanol to weight differences. The spreadsheets are saved so the data can be transferred electronically to the LIMS system.

11.3 Most samples received are accompanied with a Trip Blank (TB), In most cases the TBs are prepared by the lab and are sent along with the vials used for sample collection. The intent of the trip blank is to accompany the sample vials through all collection, preservation, shipping, and storage procedures. The infusion of outside contamination in the trip blank is not common, but can be an indicator of incorrect preparation/sampling procedures or inadequate sample storage.

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 2. is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

13.0 Calibration and Standardization

13.1 To facilitate appropriate separation and provide adequate sensitivity, the entire operating system must be correctly set up and maintained before calibration and analyses can occur. Proper settings and programming of the GC/MS volatile system greatly increase the likelihood that calibrations will be acceptable. Generating and reproducing results will also be affected favorably in a well-maintained system.

13.1.1 The following tables provide instrument settings for the daily use of the Archon/Tekmar Purge and Trap Systems. Any modifications are noted in the specific instrument's maintenance log:

PARAMETER SETTINGS FOR TEKMAR 2000/3000	
Standby	≈30° C
Preheat	40° C (low level soils only)
Purge	11.00 minutes
Dry Purge	2.0 minutes
Desorb Preheat	245° C
Desorb	2.00 minutes at 250° C

Bake	12.00 minutes at 260 ° C
Auto Drain	Off
Valve	150 ° C
Line	150 ° C
Mount	40 ° C
Gas	Helium
Flow	35 ml/minute

13.1.2An example of the GC temperature program for the SPB/ZB-624 columns used for the analysis of samples is as follows:

Start temp °C	End temp °C	Rate °C/minute	Time minutes
32	32	0.0	3.0
32	155	5.0	0.00
155	200	15.0	0.00

13.1.3 The injector is a split/splitless injector operated in split mode ranging from 1:10 to 1:60. The injector temperature is 200 °C.

13.1.4 The MS detector parameters are subject to change to achieve optimum chromatography. See instrument maintenance logbook for recent changes regarding source maintenance, as well as filament and multiplier replacements. Current tune values and EM voltage settings are documented and can be found in the appropriate instrument's tuning logbook.

13.1.5 4-Bromofluorobenzene (BFB) Standard - A standard solution containing 50 ug/ml is used for the daily tune check. The BFB is directly injected onto the column in 25 to 50 ng injections (0.5 to 1.0 uL).

13.1.5.1 The GC/MS system tune must be verified at the beginning of any calibration or a sequence run and verified every 12 hours thereafter. The tuning compound is BFB which is injected directly onto the GC column The software is set up to check the tune by using the mean of three scans across the apex. Background subtraction is performed using a single scan no more than 20 scans prior to the elution of BFB. Manual scans can be checked by taking an average of scans across the BFB peak. The tuning acceptance criteria are listed below (m/z range 35-260):

Mass (m/z)	Abundance criteria
50	15 to 40% of mass 95.
75	30 to 60% of mass 95.
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95.
173	<2% of mass 174.
174	>50% of mass 95.
175	5 to 9% of mass 174.
176	>95% but <101% of mass 174.
177	5 to 9% of mass 176.

13.1.6 The preparation of working standards is routinely performed each week unless integrity is shown to be intact. All standards are assigned a unique identification number and preparations are documented in a Standards Logbook.

13.1.6.1 Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels (in most instances, seven levels are currently used) and are prepared from the working standard dilutions of stock standards. One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels shall correspond to the expected range of concentrations found in real samples and shall contain each analyte for detection by this method. For low-level soil calibrations, sodium bisulfate is added at a 0.2 g/1.0 g sample to the water to match sample matrix /acidity if the samples were collected and preserved with sodium bisulfate. Med/high-level soil calibrations have MeOH added at a 0.1 ml /5.0 ml H2O to match sample matrix/preservation. All final concentrations are brought to volume with DI water. The following tables outline the preparation of a seven level calibration curve for water and soil samples (all calibration standards are transferred into 40 ml VOA vials for placement on the autosampler):

1) Waters Curve (5.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV and SSTD (in uL)	Final Volume (ml) (Volumetric flask)
1.0	1.0	100
2.0	2.0	100
5.0	5.0	100
10.0	10.0	100
20.0	20.0	100
30.0	30.0	100
40.0	40.0	100

2)Waters Curve (25.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV and SSTD (in uL)	Final Volume (ml) (Volumetric flask)
0.2	1.0	500
0.4	2.0	500
1.0	1.0	100
2.0	2.0	100
4.0	4.0	100
6.0	6.0	100
8.0	8.0	100

3) Low Level Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV and SSTD (in uL)	Grams of sodium bisulfate added (if needed)	Final Volume (ml) (Volumetric flask)
0.001	1.0	1.0	100
0.002	2.0	1.0	100
0.005	5.0	1.0	100
0.010	10.0	1.0	100
0.020	20.0	1.0	100
0.030	30.0	1.0	100
0.040	40.0	1.0	100

4) Medium/High Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV and SSTD (in uL)	uL of MeOH added	Final Volume (ml) (Volumetric flask)
0.050	1.0	998	100
0.100	2.0	996	100
0.250	5.0	990	100
0.500	10.0	980	100
1.000	20.0	960	100
1.500	30.0	940	100
2.000	40.0	920	100

13.1.6.2 Internal Standards - The internal standards used are Chlorobenzene-d_5, 1,4-Difluorobenzene, 1,2-Dichloroethane-d_4 and Fluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. The Archon autosampler automatically adds approximately 1 uL of the 80 ug/ml-working standard (exact amounts are determined and recorded in the maintenance logbook) to each sample/standard purged.

13.1.6.3 Surrogate Standards - The surrogate standards used are Toluene-d_8, 1,2-

Dichloroethane-d_4, 4-Bromofluorobenzene, and Dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. The 100 ug/ml-working standard is used for calibration and is added at the same concentrations as the target compounds (see above).

13.1.6.4 ISTD/SSTD Combined Standard - A combination of internal standard and surrogate standard at 80 ug/ml is automatically added by the autosampler to all samples, blanks, CCV's and spikes used for any given sequence after calibration. Limits are generated internally or project/program limits are used.

13.1.6.5 Calibration curves are prepared fresh from newly made working standards to ensure accurate concentrations are maintained.

13.1.6.6 Secondary dilution standards (when necessary) - secondary dilution standards containing the compounds of interest (usually at 10.0/100 ug/ml) for low level and MDL analyses shall be prepared in methanol and must be stored with minimal headspace and shall be checked frequently for degradation. They are to be stored for one week only.

13.1.6.7 Preparation of standards is documented in the Volatile standards logbook. Each standard solution is documented with the standard name, concentration, preparation date, expiration date and a unique number given to that standard for future traceability.

13.2 The curve is generated using the relative response factor (RRF or RF). The data system tabulates the area response of the characteristic ions against the concentration of each compound and each internal standard. Calculate response factors for each compound relative to one of the internal standards. The internal standard selected for the calculation for the RF for a compound is the internal standard that has a retention time closest to the compound being measured. The RF is calculated by the data system as follows:

$$RF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

where:

A_s = Area of the characteristic ion for the compound being measured in the calibration standard.

A_{IS} = Area of the characteristic ion for the specific internal standard.

C_{IS} = Concentration of the specific internal standard.

C_s = Concentration of the compound being measured in the calibration standard.

13.2.1 The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C. Sec. 11.5.1).

13.3 When ARFs are not acceptable, results are sometimes calculated using linear (1st order) regression curves and/or quadratic (2nd order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5.2 and sec. 11.5.3).

13.4 A system performance check shall be made before the calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are used for system performance check. These compounds are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are as follows (Method 8260B sec. 7.3.5):

	Minimum RF	Comment
Chloromethane	>0.10	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	>0.10	This compound is one of the compounds most likely to purge poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
Chlorobenzene	>0.30	
1,1,2,2 Tetrachloroethane	>0.30	The response of this compound is degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.
1,1 Dichloroethane	>0.10	The response of this compound is also degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

13.5 The RSD for the RFs for each individual calibration check compound (CCC) must be less than 30%. For the calibration to be valid, this criterion must be met for each CCC compound even if it is not a target compound for the project of concern or for the samples being analyzed. The CCC compounds are Vinyl chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, and Ethylbenzene. If the RSD of any CCC is greater than 30%, then corrective action must be taken and the system re-calibrated.

13.5.1 If the RSD of the RFs is less than 15%, then the RF is assumed to be constant over the calibration range, and the average response factor may be used for quantitation. If the RSD of any analyte or surrogate mean RF exceeds 15% but is less that 30% than linear regression or second order curves may be used for quantitation. If the RSD of the RFs is greater than 30%, either analyze additional aliquots of appropriate calibration solutions to obtain an acceptable RSD of RFs over the entire concentration range, or take action to improve GC/MS performance. Surrogate compounds are present at the same concentration on every sample, CCV, LCS, MS,

MSD and all types of blanks.

13.6 All calibrations are confirmed by the analysis of a "second source" Initial Calibration Verification (ICV) standard before daily checks and analyses are performed. The criteria for the SPCCs are the same as stated above (sec.13.3), but the CCCs must be ≤ 20 % RSD. If these criteria are not met and a reanalysis of the ICV confirms the nonconformities, then corrective actions must be taken and the instrument recalibrated. The RSD limit for all other target compounds is also 20% unless specified differently by any other applicable program or project's criteria (QSM: ±25% D for all analytes). Any outliers suggest a problem and poor performers shall be addressed. The concentrations of the ICV are near the mid point of the curve (10/100 ug/L for water-5 ml purge, 2.0/20 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The preparation of ICV's is as follows:

Water (5 ml purge)--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge)-- Spike 50 ml of DI water(volumetric flask) with 1.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 uL of this to 5.0 ml of DI water and transfer into a VOA (containing a stir bar) for analysis.

Med/high-level soils-- Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer into a VOA vial for analysis.

13.7 Finally an Initial Calibration Blank (ICB) is analyzed to confirm that the instrument is free from contamination. Any detects in the ICB shall be less than the method detection limit and/or less than ½ the program/project limits. Any detects above MDL or program limits must be addressed before sample analyses begin. To prepare an ICB fill a 40 ml VOA vial preserved with 5% HCL with DI water.

13.8 Demonstration and documentation of an acceptable initial calibration is required before any samples are analyzed. Refer to EPA SW-846, Method 8000B, Section 7, for a detailed discussion of calibration procedures.

14.0 Procedure

14.1 Prior to sample analysis a GC/MS tune and calibration check must be analyzed. Verify the MS tune and initial calibration at the beginning of each 12-hour work shift during which analyses are performed.

14.1.1 Introduce into the GC (by direct injection) 25 to 50 ng of BFB and acquire a mass spectrum that includes data for m/z 35-260. If the spectrum does not meet all criteria, the MS must be retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.

14.1.2 The calibration curve integrity for each analyte must be confirmed with the use of a CCV standard once every 12 hours of analysis time. The CCV standard is prepared at concentrations near the midpoint of the calibration curves (10/100 ug/L for water-5 ml purge, 2.0/20 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The QSM requires the CCV's to be varied throughout an Analytical run. Typically the concentrations used are 10, 20, and 30 ppb for Low Level soil and water-5 ml purged; 0.5, 1.0 1.5 mg/kg for MeOH preserved soil; and 1.0, 2.0, and 4.0 ppb for water-25 ml purged (preparation procedures are the same as listed 14.1.3, using the appropriate amount of the 100/1000 ug/ml CCV Std).

14.1.3 The CCV is placed on the autosampler in the same manner as the samples (sec. 14.2.3). The SPCC and CCC criteria must be met before the calibration is considered still valid. Preparation of CCV's is as follows:

Water (5 ml purge)--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge)-- Spike 50 ml of DI water(volumetric flask) with 1.0 uL of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml CCV standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 uL of this to 5.0 ml of DI water and transfer into a VOA vial (containing and a stir bar) for analysis.

Med/high-level soils-- Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 uL of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis.

14.1.3.1 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hour shift. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration (sec. 13.3). If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column and active sites in the column or chromatographic system.

14.1.3.2 Calibration Check Compounds (CCCs) - After the system performance

check is met, CCCs are used to check the validity of the initial calibration. If the percent difference for any compound is greater than 20%, the laboratory shall consider this a warning limit (SMFs need to be checked, as well as program/project criteria met before analysis continues). If the percent deviation for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% D.) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new calibration must be generated. This criterion must be met before quantitative sample analysis begins. If the CCCs are not required analytes by the permit, then all required analytes must meet the 20% difference criteria.

14.1.3.3 The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the average from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCV's and associated samples while the system was malfunctioning is necessary.

14.2 Sample Introduction and Purging

14.2.1 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples. Currently 18-19 purged samples including QC can be analyzed within 12 hours of the BFB injection. The Archon autosampler can be programmed to accommodate the number of samples needed per analytical shift.

14.2.2 After the continuing calibration is verified, the system must be proven to be free of contamination by analyzing a Method Blank (MB). The MB shall not contain detects above the detection limits for any given compound. Some programs allow detects up to but not exceeding one half the Method Reporting Limits (MRLs). If the method blank contains detects above the detection limits or RL's, then corrective actions must be performed to ensure the system is free from contamination; all affected samples shall also reanalyzed. The MBs are also placed on the autosampler in the same manner as the samples (sec. 14.2.3).

14.2.3 Analysis of samples begins by allowing the sample to come to ambient temperature prior to analysis. The VOA vials containing the water samples are placed on the autosampler where a 5.0 to 25.0 ml aliquot is withdrawn from the vial and added into the appropriate purge vessel. The same procedure is followed for methanol preserved soils (1.0 ml of soil extract/50.0 ml DI H2O is prepared and added into a 40 ml VOA vial prior to adding the samples to the autosampler). Low level soils are prepared by adding the VOA vial containing a magnetic stir bar and ≈5 g sample/5.0 ml DI H2O to the autosampler (2-5 grams of sample required for low-level analysis). The autosampler then adds an additional 5.0-mL of H2O containing the ISTD/SSTD mixture. The sample is heated to 40°C and

purged in the VOA vial while being stirred, and the volatiles are collected onto the trap.

14.2.3.1 The ISTD/SSTD is added automatically by the Archon autosampler as the sample is transferred from the 40 ml sample vial to the sparge tube; the exception is for low level soils as noted above (sec.14.2.3).

14.2.3.2 The sample is purged for 11 minutes at ambient temperature (40°C for low-level soils) using helium with a flow of 35 ml/min at a 20 psi purge pressure.

14.2.3.3 During the 11-minute purge time, the purgeable volatile organics are adsorbed onto the Supelco Carbosieve K trap.

14.2.3.4 During desorbtion the trapped materials are rapidly heated while back-flushing the trap with helium at 35 ml/min. for 2 minutes at 250°C and introduced in the GC/MS. After the valve to the GC is closed the trap is then baked and back flushed with helium for ≈12 minutes at 260°C.

14.2.3.5 The GC is temperature programmed at 32°C for 3 minutes, then ramped to 155°C at 5°C/min, and finally ramped to 200°C at 15°C/min. The column flow is set at 1 ml/min. constant flow using helium as the carrier gas.

14.2.3.6 The transfer line to the MS is maintained at 250°C and the ion source is maintained at ≈260°C while under constant vacuum. The GC injector is set at 200°C.

14.2.4 For each sample batch a matrix spike (MS), matrix spike duplicate (MSD), and laboratory control spike (LCS) is prepared and analyzed. The concentrations for water spikes are 10.0/100 ug/L for 5 ml purge and 1.0/10.0 ug/L for 25 ml purge. The spiked concentrations for soil samples are ≈0.010/0.10 mg/kg for low level and ≈0.50/5.0 mg/kg for MeOH preserved depending on sample weights and percent solids. One exception is for the analysis of field collected MeOH preserved soils. These samples are analyzed with a laboratory control spike and a laboratory control spike duplicate (LCSD). All spikes are transferred into 40 ml VOA vials and added to the autosampler. The spike concentrations may vary depending on program/project specific criteria, but the preparation volumes are constant and only the spiking amount changes. 14.2.4.1 lists examples of spike preparation based on the concentrations above.

14.2.4.1 The preparation of the matrix spikes is performed as follows:

Water (5 ml purge)--Spike 40 ml of sample with 4.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer to a VOA vial for analysis. When adequate sample amounts are not provided, one 40 ml aliquot of sample is spiked and split into two separate VOA vials containing 15 ml glass inserts.

Water (25 ml purge)-- Spike 50 ml of sample with 1.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer into a VOA vial for

analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/100 ug/ml ICV standard, invert three times and transfer 5.0 ml into a VOA vial containing ≈5 g of sample and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 uL of this to 5.0 ml of DI water and transfer into a VOA vial containing ≈5 g of sample and a stir bar for analysis.

Med/high-level soils--Spike ≈10 g of sample contained in a VOA vial with 50 uL of the 100/1000 ug/ml ICV standard. Add 9.95 ml of methanol to the spiked sample and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis.

14.2.4.2 The preparation of laboratory control spikes is performed as follows:

Water (5 ml purge)--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge)-- Spike 50 ml of DI water (volumetric flask) with 1.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 uL of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

Med/high-level soils--Spike 10 g of control sand contained in a VOA vial with 50 uL of the 100/1000 ug/ml ICV standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of
methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis.

14.2.5 The data is collected by the Chemstation software using the response factors (or linear/second order regressions when necessary), and results are calculated using the internal standard method of quantitation. Response factors for each detected compound are compared with that obtained in calibration, and based on those comparisons, results are generated. Software manuals define the procedures for creating and understanding a specific method (Understanding Your Chemstation, Hewlett Packard, G2070-90100, October, 1994, Environmental Forms Software, Hewlett Packard, G1032-90021, November, 1992, and Productivity Enhancement Software for HP Chemstation, Prolab Resources Inc., XMS01A-002, Rev. G, 2001).

15.0 Calculations

15.1 The Chemstation software (using response factors) calculates the initial concentration (or raw result) of target compounds as follows:

Liquids

$$\text{Initial concentration } (\mu g/L) = \frac{A_X \times I_{IS}}{A_{IS} \times RF}$$

Where:

A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (ug/L). Typical concentrations used are 20.0 ug/L for 5.0 ml purge, and 4.0 ug/L for 25.0 ml purge
A_{IS}= Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured.

Solids

$$\text{Initial concentration (mg/kg)} = \frac{A_X \times I_{IS}}{A_{IS} \times RF}$$

Where:

A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (mg/kg). Typical concentrations used are 0.020 mg/kg for low-level soils and 1.0 mg/kg for med/high-level soils.
A_{IS}= Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured.

15.2 The Chemstation software (using linear regression) calculates the initial concentration of target compound as follows:

Response Ratio = slope * amount ratio + intercept

Where: Response Ratio = response of target compound / response of associate ISTD
Amount Ratio = target compound conc. / associated ISTD conc.

Example: Tr / ISr = m * Tc / ISc + b

Where: Tr = response of target compound.
ISr = Internal Standard response
m = slope of the curve (for the target compound)
ISc = Internal Standard concentration
Tc = Target compound concentration
b = y intercept of the curve (for the target compound)

Solve for "Tc"

15.3 The initial concentration results are then transferred to the laboratory's LIMS system where the final concentrations are calculated.

15.3.1 The final concentration for water samples is calculated as follows:

Final concentration (ug/L) = Initial concentration x DF

Where:

DF = Dilution Factor

15.3.2 The final concentrations for low-level and med/high-level soils are calculated by the following equation:

$$\text{Final concentration (mg/Kg)} = \frac{\text{Initial concentration x Sample volume x DF}}{\text{Sample weight x Percent solids}}$$

Where:

Sample volume = 5.0 ml for low-level soils, or volume of MeOH used for med/high-level soils preservation.

Sample weight = grams of sample place in VOA vial for low-level soils, or total grams of sample preserved for med/high-level soils.

Percent solids = fraction equivalent (e.g. 97.1 % = 0.971)

DF = Dilution factor (applicable for MeOH preserved soils).

15.3 The spike percent recoveries (%R) and relative percent differences (RPD) are calculated in LIMS as follows:

Liquids--concentration (conc.) of spike added:

$$\text{ug/L} = \frac{\text{ml of spike added x conc. spiking std..}}{\text{ml of sample (or DI H2O) spiked}} \times 100$$

Solids--concentration of spike added:

$$\text{mg/kg} = \frac{\text{ml of spike added x conc. spiking std..}}{\text{grams of sample (or control sand) spiked.}}$$

$$\text{MS/MSD \%R} = \frac{\text{(conc. spike obtained – conc. Sample obtained)}}{\text{conc. spike added}} \times 100$$

$$\text{MS/MSD RPD} = \frac{[\text{conc. MS} - \text{conc. MSD}]}{(\{\text{conc. MS} + \text{conc. MSD}\}/2)} \times 100$$

$$\text{*LCS \% R} = \frac{\text{conc. spike obtained}}{\text{conc. spike added}} \times 100$$

Notes:
--Concentrations (conc.) of samples, MS/MSD, and LCS spikes are obtained directly from calibration curve.
--Soil spike concentrations and recoveries are calculated on a dry weight basis.
--[ ] Signifies absolute values.
--* Equation can also be used to calculate surrogate recoveries

16.0 Method Performance

16.1 Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

16.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in μg/L and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000B, sec. 8.4.9).

16.2.1 Examples of the preparation of IDCs are as follows:

Water (5 ml purge)--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge)-- Spike 50 ml of DI water(volumetric flask) with 1.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 uL of the 100/1000 ug/ml ICV standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 uL of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

Med/high-level soils--Spike 10 g of control sand contained in a VOA vial with 50.0 uL of the 100/1000 ug/ml ICV standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis.

16.3 Many programs (i.e. USACOE) require the analysis of method reporting limit (MRL) standards and method detection limit (MDL) check samples as another means of checking method performance. The MRLs are analyzed at the beginning and end of each 12 hour shift and are typically prepared at concentrations equal to the lowest standard on the calibration curve. Recovery limits are program specific but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x the method detection limit. The MDL check sample is analyzed quarterly (as a minimum) to confirm instrument sensitivity (e.g. to verify that the method detection limits are still achievable). The MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). In most instances, a method detection limit check sample is analyzed at the end of each sequence requiring an MRL standard. The recovery criteria for MDL check samples are the ability to detect all compounds. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

16.3 Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SSTD, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Other programs such as the Shell Document for the USACOE use default LCS limits of 80-120 % recovery for water samples and 75-125 % for soil samples (SMF 60-140%). The Shell also uses default MS/MSD recovery limits for water and soils of 70-130% (SMF 60-140 %), and default precision limits of 30% for water samples (SMF 40%, no RPD limits for soils). The WI UST program uses default accuracy and precision limits for surrogates and spikes of 80-120/20 %. Client and/or Project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 2.) also lists recovery limits specific to the method/project/program.

17.0 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address their waste generation.

17.2 The quantity of chemicals purchased shall be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes shall reflect anticipated usage and reagent stability.

18.0 Data Assessment & Acceptance Criteria for QC Measures

18.1 If the initial analysis of a sample or a dilution of the sample has a concentration of an particular analyte that exceeds the calibration range, the sample must be reanalyzed at a

dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analyses can not resume until a blank can be analyzed that is free of interferences.

18.2 After the analyses of water samples, the pH shall be taken to verify proper field preservation. pH strips are used to verify the pH which is then documented in the bench sheet logbook.

18.3 Qualitative Analysis (Method 8260B, sec. 7.6)

18.3.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum (ion scans) after background correction with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated (by the laboratory) using the conditions of this method. The mass spectral library is updated with each new calibration and is continually updated with the mass spectra from CCV's.

18.3.2 The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity or any ions over 30% relative intensity if fewer than three such ions occur in the reference spectrum. Table 3 lists compounds along with the Primary Ion (Quantitation ion) used for calculating results, and the Secondary Ions (Qualitative ions) used for qualitatively matching sample spectrums with reference spectrums for positive identifications. Compounds shall be identified as present when the criteria below are met.

18.3.2.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

18.3.2.2 The relative retention time (RRT) of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

18.3.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

18.3.2.4 Structural isomers that produce very similar mass spectra shall be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

18.3.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co eluting compound.

18.3.3 For samples containing compounds that are not a part of the normal target list, a library search may be required for the purpose of tentative identification. Tentative identified compounds (TICs) are needed only when requested or required by a particular project or program. Data system library search routines shall not use normalization routines that would misrepresent the library of unknown spectra when compared to each other. Use the following a guidance for reporting TICs (Method 8260B, sec. 7.6.6):

1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) shall be present in the sample spectrum

2) The relative intensities of the major ions agree within ± 20%.

3) Molecular ions present in the reference spectrum shall be present in the sample spectrum.

4) Ions present in the sample spectrum but not in the reference spectrum shall be checked for possible background contamination. They shall also be reviewed for possible co elution with another compound.

5) Ions present in the reference spectrum but not in the sample spectrum shall be check against the possibility of subtraction from the sample spectrum due to background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies.

18.4 Quantitative Analysis (Method 8260B, sec. 7.7)

18.4.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is performed by the data system using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte. Quantitation is performed using the RF averages from the initial (i.e. 7 pt) calibration, and not the continuing calibration check (ICV).

18.5 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the reference spectrums are compared and the sample spectrums and identifications have been made, the sample data can be reported.

Assessments of all spiked and calibration control samples and standards shall also be finalized before reporting the data.

18.5.1 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (Table 4.) is filled out and signed confirming that all the data has been thorough scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs his or her portion of the checklist (an example of the checklist is included in this SOP). Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

18.5.2 A paper hard copy of the data is then filed or archived. The package includes the checklist, the sequence run log, a copy of the bench sheet, the LIMS run log, verification of tuning and system performance data, and verification of calibration data. For each sample, the chromatogram, quantitation and library spectra (ion scans) for all positive target compounds are also included. All the data is to be initialed and dated by the analyst. Each data file header shall contain the sample ID # and the date and time acquired.

19.0 Corrective Measures for Out-of-Control Data

19.1 When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination, and the sample may need diluting to reduce matrix affect. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions are to be taken for different QC-type failures or nonconformities (Table 2.).

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

20.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.

20.1.1 The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst shall determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Loss of data due to downed or malfunctioning instrumentation can be addressed with the use of backup instrumentation. If an instrument becomes unusable, the samples shall be analyzed on a different

instrument system. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

20.1.2 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more sample can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

20.1.2.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

20.1.2.2 In all instances, if results are reported from data that is out of control or unacceptable, that data must be qualified accordingly. Once the client has been notified and he or she instructs us to report the data, then flag the data indicating what type of nonconformity has occurred.

20.1.2.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder shall be labeled as such, indicating that the data is out of control.

20.1.2.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

21.0 Waste Management

21.1 Samples are routinely held (refrigerated) for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

22.0 References

22.1 USEPA, SW-846 Methods 8000C, Rev. 3, March, 2003
22.2 USEPA, SW_846 Method 8260B, Rev. 2, December, 1996
22.3 USEPA, SW-846 Methods 5030B, Rev. 2, December, 1996
22.4 USEPA ,SW-846 Methods 5035 (inc. App. A), Rev. 0, December, 1996
22.5 National Environmental Laboratory Accreditation Program (NELAP), Quality Systems, Chapter 5, June, 2000
22.6 USEPA, Method 603, Acrolein and Acrylonitrile, July, 1982
22.7 Department of Defense, Quality Systems Manual for Environmental Laboratories, DoD Environmental Data Quality Workgroup,

Department of Navy, Lead Service, Draft Version 3, December 2004.

22.8 Louisville Chemistry Guideline (LCG), US Army Corps of Engineers-Louisville District, June 2002.

22.9 Louisville DOD Quality Systems Manual Supplement (LS), US Army Corps of Engineers-Louisville District, March 2007.

Table 1.
Analyte List

Analyte	Analyte
Acetone	2,2-Dichloropropane
Benzene	1,1-Dichloropropene
Bromobenzene	cis-1,3-Dichlropropene
Bromochloromethane	trans-1,3-Dichloropropene
Bromodichloromethane	Diisopropyl ether
Bromoform	Ethylbenzene
Bromomethane	Hexachlorobutadiene
2-Butanone	2-Hexanone
n-Butylbenzene	Isopropylbenzene
sec-Butylbenzene	p-Isopropyltoluene
tert-Butylbenzene	Methylene chloride
Carbon disulfide	4-Methyl-2-pentanone
Carbon tetrachloride	Methyl tert butyl ether
Chlorobenzene	Naphthalene
Chloroethane	n-Propylbenzene
2-Chloroethylvinyl ether	Styrene
Chloroform	1,1,1,2-Tetrachloroethane
Chloromethane	1,1,2,2-Tetrachloroethane
2-Chlorotoluene	Tetrachloroethene
4-Chlorotoluene	Tetrahydrofuran
Dibromochloromethane	Toluene
1,2-Dibromo-3-chloropropane	1,2,3-Trichlorobenzene
1,2-Dibromoethane	1,2,4-Trichlorobenzene
Dibromomethane	1,1,1-Trichloroethane
1,2-Dichlorobenzene	1,1,2-Trichloroethane
1,3-Dichlorobenzene	Trichloroethene
1,4-Dichlorobenzene	Trichlorofluoromethane
Dichlorodifluoromethane	1,2,3-Trichloropropane
1,1-Dichloroethane	1,2,4-Trimethylbenzene
1,2-Dichloroethane	1,3,5-Trimethylbenzene
1,1-Dichloroethene	Vinyl chloride
cis-1,2-Dichloroethene	Vinyl acetate
trans-1,2-Dichloroethene	o-Xylene
1,2-Dichloropropane	m/p-Xylene
1,3-Dichloropropane	

Table 2.
Volatile Organic Compounds by GC/MS
Summary of Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (BFB)	Every 12 hours.	Ensure correct mass assignment. BFB % Relative abundance criteria as specified in method 8260 or use program/project specific criteria.	Retune. Do not proceed with analysis until tune meets criteria.
Initial Calibration (ICAL)	Each time the instrument is set up and when CCCs and SPCCs in the continuing calibration verification (CCV) do not meet criteria. Established initially at minimum five concentration levels (six concentration levels if a second order {quadratic} curve is used) - low standard at or below project required reporting limit (PRRL), near but above method detection limits (MDL). Heated purge for low-level soils.	1. Average relative response factors (RRFs) for SPCCs Chloromethane, 1,1-Dichloroethane, and Bromoform ≥0.10 and for SPCCs Chlorobenzene and 1,1,2,2-Tetrachloroethane ≥ 0.30. 2. % RSD for RRFs for each CCC ≤30%. 3. % RSD for RRFs for all target compounds ≤15%. IF RF % RSD >15% use linear curve, r =.995, r2 = .990. 4. LCG, NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents	Correct system and recalibrate. Criteria must be met before sample analysis may begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	Every 12 hours. Shall be at or near the mid-point of calibration range for all target compounds, CCCs, SPCCs and surrogates and is prepared from second source standards. Typically use 10/100 ppb for H2O and Low Level Soils, 0.5/5.0 mg/kg for MeOH preserved soils. Heated purges for low-level soils.	1. RRF for SPCCs Chloromethane, 1, 1-Dichloroethane, and Bromoform ≥0.10 and for SPCCs Chlorobenzene and 1,1,2,2-Tetrachloroethane ≥0.30. 2. % Deviation. for RRFs of each CCC <20%. 3. Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to 10 % of target compounds = 40% RSD. 4. LCG, NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents (ex: ± 25% D. for QSM projects).	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % relative standard deviation (RSD) >20%, then system must be inspected and problem corrected before sample analysis. If >20% RSD then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. ACOE allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Continuing Calibration Verification standards(CCV)	Every 12 hours. Shall be at or near the mid-point of calibration range for all target compounds, CCCs, SPCCs and surrogates and is prepared from standards used for calibration (Typically use 10/100 ppb for H2O and Low Level soils, 0.5/5.0 mg/kg for MeOH preserved soils). Varied CCV levels are required for QSM when multiple CCV's are necessary on a run (Typically use 10, 20, and 30 ppb for H2O and Low Level soils, 0.5, 1.0, and 1.5 mg/kg for MeOH preserved soils). Heated purges for low-level soils..	1. RRF for SPCCs Chloromethane, 1, 1-Dichloroethane, and Bromoform ≥0.10 and for SPCCs Chlorobenzene and 1,1,2,2-Tetrachloroethane ≥0.30. 2. % Deviation for RRFs of each CCC <20%. 3. Non-CCCs - <20% Deviation for RRFs, <20 % Drift for linear curve and non linear curves- Sporadic marginal failure of up to 10 % of target compounds = 40% RSD. 4. LCG, NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % RSD >20%, then system must be inspected and problem corrected before sample analyses. If >20% RSD correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. In any case sample results reported that have %D failures must be qualified (A). ACOE allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in associated CCV standard. 2. Retention time (RT) within 30 sec of RT for associated CCV standard. 3. LCG, NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Reanalyze sample. * If reanalysis is outside limits the data shall be qualified (S). Follow specified criteria as stated in Shell or other documentation.
Method Blank (MB)	1 / 20 samples per matrix or at contact/ program specific frequencies. The MB is used to document contamination resulting in the analytical process and shall be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern shall be less than the highest of either : *Method Detection Limit *Five percent of the regulatory limit for that analyte or, *Five percent of the measured concentration in the sample. 2. ACOE/QSM: ≤ ½ MRL 3. Follow criteria according to specific program/agency.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples.* For ACOE/QSM data if less than ½ MRL no action required.* *If reanalysis of blank still contains contamination above specified limits, affected data shall be flagged (B).

Laboratory Control Sample (LCS)	1 / 20 samples or at contact/ program specific frequencies. Must undergo all sample preparation procedures. Prepared from a second source and contain target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries (and RPDs, if applicable) within in-house generated limits. Default 70-130% (20% RPD). Sporadic marginal failure (SMF) of up to 10 % of target compounds = 60-140 % rec. 2. Use QSM, LCG, project specific, or client contract limits when applicable.	If LCS recoveries are within control limits or within SMF frequency and limits then no action is required. If the LCS exceeds control limits, as well as SMF criteria the reanalyze the LCS to confirm proper preparation procedure. If still exceeding limits then reanalyze associated samples with a new LCS.. If sample data is reported with LCS failures then that data must be qualified (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.
Matrix Spike/Matrix Spike Duplicate	One per set of 20 samples per matrix. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries and RPDs within in-house generated limits. Default 70-130 / 20%RPD. 2. Use QSM, LCG, project specific, or client contract limits when applicable.	If LCS is acceptable, then report probable matrix interference. Qualify non-detects if the recoveries are low (M), and detects if the recoveries are low and the sample amount + the true spiked amount shall be within calibration range. Qualify detects if recoveries are high and the detects + the true spiked amount shall be within calibration range. If recoveries are high and there are no detects in the unspiked sample then that data does not require flagging. If spiked amount + sample amount for any given compound exceeds calibration range than the spike is considered invalid for that compound. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.

Qualitative/Quantitative Issues	1. If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed. 2. If the concentration of the target analyte (that exceeded the calibration range) is present in the sample following the high level sample and is greater than the RL but ≤5x RL, then that sample must be reanalyzed to determine if carryover occurred.	1. The instrument level of all compounds must be within the calibration range for all samples. 2. The sample analyzed immediately after a high-level sample must display concentrations of the high level target compounds less than the RL or greater than 5x RL.	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results shall be flagged (X). A sample displaying concentrations of target compounds between the RL and 5x the RL that was analyzed immediately after a high-level sample must be reanalyzed. If the results do not agree within the RL, report only the second analysis.
Surrogate	1. Calibrated as target compounds. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. All % Recoveries within in-house generated limits. Default 70-130%. 2. Use QSM, LCG, project specific, client contract limits when applicable.	If recoveries are not within limits: Check to be sure that there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance. If no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (S). Due to matrix affect, no reanalysis is required if the MS and/or MSD are outside limits.

Sample Duplicate (Dup) – when required	1. Program/contract specific. 2. When limited sample is available a sample duplicate may be used in leu of a MSD.	1. RPD < or = 10% (between sample and sample duplicate) for QSM projects. 2. RPD's within in-house limits. Default ± 20%. 3. QSM, LCG, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	If RPDs are not within limits: Check to be sure that there are no errors in calculations. Also, check instrument performance and correct if necessary. If corrected or no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (Y).
Method Reporting Limit (MRL) Spike – when required	1. Program/contract specific. 2. Typically bracketing samples for every 12 hour analysis window.	1. % Recoveries within in-house generated limits. Default 70-130 %Rec. 2. Program or project/contract specific limits shall be followed as stated in their documents..	If there is a failure investigate problem. If system is in control run an MDL check sample to verify detection limits.

Table 3.
Characteristic ions

Analyte	Primary Ion	Secondary Ion(s)	Analyte	Primary Ion	Secondary Ion(s)
Acetone	43	58	2,2-Dichloropropane	77	97,79
Benzene	78	51,77	1,1-Dichloropropene	110	77,75
Bromobenzene	156	77,158	cis-1,3-Dichlropropene	75	110
Bromochloromethane	128	49,130	trans-1,3-Dichloropropene	75	77,110
Bromodichloromethane	83	85,129	Diisopropyl ether	45	87,43
Bromoform	173	175,171	Ethylbenzene	91	106
Bromomethane	94	96	Hexachlorobutadiene	225	223,227
2-Butanone	43	72,57	2-Hexanone	43	58,57
n-Butylbenzene	91	92,134	Isopropylbenzene	105	120
sec-Butylbenzene	105	134	p-Isopropyltoluene	119	134,91
tert-Butylbenzene	119	91,134	Methylene chloride	84	86,49
Carbon disulfide	76	78	4-Methyl-2-pentanone	43	58,57
Carbon tetrachloride	119	121	Methyl tert butyl ether	73	57,43
Chlorobenzene	112	77,114	Naphthalene	128	51,129
Chloroethane	64	66	n-Propylbenzene	91	120
2-Chloroethylvinyl ether	63	65,106	Styrene	104	78
Chloroform	83	85	1,1,1,2-Tetrachloroethane	131	133,119
Chloromethane	50	52	1,1,2,2-Tetrachloroethane	83	85
2-Chlorotoluene	91	126	Tetrachloroethene	166	168,129
4-Chlorotoluene	91	126	Tetrahydrofuran	42	72,71
Dibromochloromethane	129	127,131	Toluene	92	91
1,2-Dibromo-3-chloropropane	157	155	1,2,3-Trichlorobenzene	180	182,145
1,2-Dibromoethane	107	109	1,2,4-Trichlorobenzene	180	182,145
Dibromomethane	93	95,174	1,1,1-Trichloroethane	97	99,61
1,2-Dichlorobenzene	146	111,148	1,1,2-Trichloroethane	83	97,85,99
1,3-Dichlorobenzene	146	111,148	Trichloroethene	95	130,132
1,4-Dichlorobenzene	146	111,148	Trichlorofluoromethane	101	103,105
Dichlorodifluoromethane	85	87	1,2,3-Trichloropropane	75	110
1,1-Dichloroethane	63	65,83	1,2,4-Trimethylbenzene	105	120
1,2-Dichloroethane	62	98,64	1,3,5-Trimethylbenzene	105	120
1,1-Dichloroethene	96	61,63	Vinyl chloride	62	64
cis-1,2-Dichloroethene	96	61,98	Vinyl acetate	43	86
trans-1,2-Dichloroethene	96	61,98	o-Xylene	106	91
1,2-Dichloropropane	63	76,112	m/p-Xylene	106	91
1,3-Dichloropropane	76	78			
ISTD			**SSTD**		
Fluorobenzene	96	77	Dibromofluoromethane	113	111,192
Chlorobenzene-d_5	117		1,2-Dichloroethane-d_4	102	104
1,4-Dichlorobenzene-d_4	152		Toluene-d_8	98	100
			4-Bromofluorobenzene	95	174,176

Table 4. Review Checklist

INDEPENDENT DATA REVIEW CHECKLIST			Method: GCMS (EPA SW-846 8260)		
Analysis Date	Analyst/Data Interpreter	Independent Reviewer	Date or Review	Approved?	
				Yes	No
Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.					

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments:
		Yes	No	Yes	No	
I. BFB Tune Check						
BFB tune check analyzed?	Relative abundance criteria met?					
II. Initial Calibration						
Was initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MDL/LOD?					
Were SPCC compounds acceptable?	Avg. RRF> 0.1; 0.3?					
Were CCC RF's less than 15% ? (Or alternatively no greater than 30% if generating results using linear regression?)						
Was Initial Calibration Blank analyzed?						
Were the ICB results for all target analytes less than the limit of detection (LOD).	<LOD or < project/program specific limits					
III. Continuing Calibration Verification (CCV)						
Was a BFB tune check run at the beginning of every twelve hour shift?	Relative abundance criteria met?					
Was a CCV analyzed after every 12 hour tune check?						
Were SPCC compounds acceptable?	Avg. RRF> 0.1; 0.3?					
Were CCC compounds acceptable?	%D < 20%					
Were all other compounds acceptable? If yes, then continue run.	%D < 20%					
If No, did number of failures exceed sporadic marginal failure limits?	<10% failing or see project/program limits					If yes, samples reanalyzed
Were detects for outlying compounds qualified?						

Requirement:	AcceptanceCriteria	AnalystReview		Independent Review		Comments:
		Yes	No	Yes	No	
IV. Blanks						
Was method blank (MB) analyzed prior to analysis of samples?						
Were the MB results for all target analytes less than the limit of detection (LOD)?	*All target analytes <LOD or < project/program specific limits					*see SOP for more detailed criteria
If analytes were detected in the MB with no associated positives in the samples, no further action is needed. If analyte detects are greater than the acceptance criteria with associated positives in the samples, flag associated positives with "B".						
V. Laboratory Control Spike (LCS)						
Was a LCS analyzed at a frequency of one per 20 samples (or program specified frequency)?						
Were the LCS recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in-house or project/program specified limits.					
If no, were detects (and non-detects for low recoveries) qualified?						
Was the LCS a second source to the initial calibration standards?						
VI. Laboratory Control Spike Duplicate (LCSD). This section applies only to runs without MS/MSD						
Was a LCSD analyzed at a frequency of one per 20 samples (or program specified frequency)?						
Were the LCSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in house or project/program specified limits.					
If no, were detects (and non-detects for low recoveries) flagged "Q"?						
Is the relative percent difference (RPD) for each analyte between duplicate samples acceptable?	Default 20%, or see in-house or project/program specified limits.					
If No, were detects in samples flagged "Y"?						
VII. Matrix Spike/Matrix Spike Duplicate (MS/MSD)						
Was a MS and a MSD analyzed at a frequency of one each per 20 samples (or program specified frequency)?						
Were the MS/MSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in-house or client specified limits.					

Requirement:	Acceptance Criteria	AnalystReview		Independent Review		Comments:
VII. MS/MSD continued						
		Yes	No	Yes	No	
If No, were non-detects for analytes with low MS/MSD recoveries and detects < 1x spiked amount flagged "M"?						
Is the relative percent difference (RPD) for each analyte between duplicate samples acceptable?	Default 20%, or see in-house or project/program specific limits.					
If No, were detects in samples <1x the spiked amount flagged "Y"?						
VIII. Sample Analyses						
Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
Were surrogate recoveries for all samples and QC within acceptance criteria?	Default 70-130%, or see in-house or project/program specified limits.					
If no, was the sample reanalyzed and/or flagged "S"?						
Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?	Target upper middle range of calibration curve.					
Did all samples meet hold time and preservation criteria as defined by method/program.						
Was a sample with detects between LOD and 5x LOD reanalyzed if following a sample with high level detects exceeding calibration range?	Detects are not attributed to carryover					
Were all samples and QC injected within 12 hours of BFB tune check?						
Were internal standard recoveries acceptable relevant to associated CCV?	Response = -50 to +200%; Ret. time = +/- 30 sec.					
IX. Records and Reporting						
Is sequence file / injection log present in the data package?						
Were all data, calculations, and values verified in LIMS upon completion of data capture?						
Were reported results with amounts exceeding acceptance criteria flagged with appropriate qualifier and, if needed, a NCR completed?						
Was the integrity of the sample kept intact? If not, was a NCR completed?						

ATTACHMENT 5
Assessment Checklists

Form: QA-001
Date: 30-Apr-09
Version 1.2

HEALTH AND SAFETY CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, or N/A). If "no" is checked, please provide an explanation on the form.

	Documentation	Yes	No	N/A
1	Is the Site Health and Safety Plan (SSHP) on the Site?	☐	☐	☐
2	Has the SSHP and/or supplement been reviewed, dated, and signed within thelast year?	☐	☐	☐
3	Are the tasks being completed reflected in the Supplement?	☐	☐	☐
4	Is there a written acknowledgement that all employees have been briefed on and read the SSHP (signature sheet)?	☐	☐	☐
5	Are the following training records current and available:			
-	40-Hour HAZWOPER for ALL employees?	☐	☐	☐
-	24 Hours Supervised Field Experience?	☐	☐	☐
-	8-Hour HAZWOPER Annual Refresher?	☐	☐	☐
-	CPR/First Aid (minimum one person on site)?	☐	☐	☐
-	8-Hour Hazardous Waste Site Supervisor and refresher?	☐	☐	☐
-	Initial Site Health and Safety Briefing?	☐	☐	☐
6	Site Health and Safety Briefing for each location or site (record in field log notebook)?	☐	☐	☐
7	Were fire extinguishers checked on first day?	☐	☐	☐
8	Were applicable Material Safety Data Sheets at the Site (located in SamplingPlan)?	☐	☐	☐
9	Are documents current and available that indicate personnel are medically fit towork and wear the required personal protective equipment (if required)?	☐	☐	☐
10	Are work zones adequately designated?	☐	☐	☐
11	Is required personal protective equipment available and correctly used, maintained, and stored?	☐	☐	☐
12	Is the following emergency equipment located at each site:			
-	Fire extinguisher?	☐	☐	☐
-	Eye wash (minimal)?	☐	☐	☐
-	Communications (walkie talkie or phone)?	☐	☐	☐
-	First aid kit?	☐	☐	☐
13	Is the buddy system in use?	☐	☐	☐
14	Are personnel refraining from drinking, chewing, smoking, taking medications, or other hand-to-mouth contact while working in the exclusion zone?	☐	☐	☐
15	Is the site organized to allow the use of lifting equipment, and avoid tripping hazards and spreading contamination?	☐	☐	☐
16	Was a random employee asked if he/she know site hazard and emergency procedures?	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

_____
_____
_____
_____
_____

Form: QA-002
Date: 30-Apr-09
Version 1.2

SAMPLE COLLECTION CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, not observed (N/O) or N/A). If "no" is checked, provide an explanation on the form.

	General	Yes	No	N/O	N/A
1	Were new protective gloves worn between sampling locations and/or intervals?	☐	☐	☐	☐
2	Were samples collected using methods described in the FSP?	☐	☐	☐	☐
3	Were sample containers filled in the correct order? (VOCs first)	☐	☐	☐	☐
4	Was sampling equipment appropriate for the purpose and site conditions?	☐	☐	☐	☐
5	Was sampling equipment decontaminated or disposable/dedicated equipment used between each sample?	☐	☐	☐	☐
6	Were procedures for collecting QA/QC samples followed as per the FSP?	☐	☐	☐	☐
7	Were sampling locations properly identified by land survey?	☐	☐	☐	☐
8	Were bottles adequately protected from contamination prior to sample collection?	☐	☐	☐	☐
	Ground Water for Chemical Analysis				
9	Were ground water parameters stable before sample collection (as per FSP)?	☐	☐	☐	☐
10	Were turbidity readings below 50 NTU (or if all other field parameters are stable and turbidity cannot be lowered below 50 NTU, were turbidity readings within + or – 10% over three, five-minute readings)?	☐	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

Form: QA-003
Date: 30-Apr-09
Version 1.2

FIELD DOCUMENTATION CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, or N/A). If "no" is checked, please provide an explanation on the form.

	Field Documentation	**Yes**	**No**	**N/A**
1	Was all original field data, except boring logs, recorded in black indelible ink?	☐	☐	☐
2	Were log books filled out properly; accurately recounting the days events?	☐	☐	☐
3	Were all field forms completed and information accurately recorded:			
-	Field Sampling Forms	☐	☐	☐
-	Chain of Custody Forms	☐	☐	☐
-	Field Log Notebooks	☐	☐	☐
-	List additional field forms completed: _____	☐	☐	☐
	_____			
4	Was field documentation forwarded to office for peer review and QC if requested?	☐	☐	☐
5	Were deficiencies reported to QC Manager/Project Team Manager?	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

_____
_____
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Form: QA-004
Date: 30-Apr-09
Version 1.2

DECONTAMINATION CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, not observed (N/O) or N/A). If "no" is checked, provide an explanation on the form.

	Packing, Storing, and Shipment of Samples	**Yes**	**No**	**N/O**	**N/A**
1	Were the samples handled according to the FSP?	☐	☐	☐	☐
2	Did the samples remain on ice or refrigerated (except for sample transfer from coolers or refrigerators) from collection until cooler was taped for shipment?	☐	☐	☐	☐
3	Were COC forms filled out accurately and completely including project name and number, sampling date, sampling time, analytical parameters, preservatives, size and number of containers for each analytical parameter, and media sampled?	☐	☐	☐	☐
4	Were COC forms signed and dated by the preparer and the form taped to the inside of the cooler lid?	☐	☐	☐	☐
5	Were signed and dated custody seals properly placed on the cooler and the cooler sealed with strapping tape?	☐	☐	☐	☐
6	Was a shipping label attached to the cooler?	☐	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

Form: QA-005
Date: 30-Apr-09
Version 1.2

INSTRUMENT CALIBRATION CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, not observed (N/O) or N/A). If "no" is checked, provide an explanation on the form.

	Instrument Calibration	Yes	No	N/O	N/A
1	Were all field instruments calibrated properly according to manufacturer's instructions?	☐	☐	☐	☐
2	Were all field instruments calibrated and calibration checked on the schedule in the FSP?	☐	☐	☐	☐
3	Did the calibration log form(s) list all calibration events?	☐	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

Form: QA-006
Date: 30-Apr-09
Version 1.2

DECONTAMINATION CHECKLIST

Project Name/Number: _____
Site Information/Location: _____
Date: _____

Answer each question by checking the appropriate column (yes, no, not observed (N/O) or N/A). If "no" is checked, provide an explanation on the form.

	Equipment	Yes	No	N/O	N/A
1	Was all sampling equipment decontaminated properly prior to use and between sample intervals?	☐	☐	☐	☐
2	Was each decontamination event recorded in the log book?	☐	☐	☐	☐
3	Was IDW (decontamination water) properly handled?	☐	☐	☐	☐

The QC Inspector shall sign this checklist upon completion of all items on the checklist.

QC Inspector Signature: _____

Notes:

7.0 Quality Control and Quality Assurance

SOP FO-12 Quality Control and Quality Assurance Checklist	
CTI Section:	Scope of Audit:
Completed by:	Date completed:
Reviewed by:	Date reviewed:

Was an internal Audit Plan prepared?	YES	NO	N/A	
Were all internal audit sections completed?	YES	NO	N/A	
Was the internal audit process evaluated?	YES	NO	N/A	
Were audit results summarized?	YES	NO	N/A	
Was an Internal Audit Report prepared?	YES	NO	N/A	
Have internal auditor(s) signed and dated each working paper?	YES	NO	N/A	
Has each working paper been reviewed and dated?	YES	NO	N/A	
Has the Internal Audit Report been signed and dated?	YES	NO	N/A	
Did auditor conduct interviews?	YES	NO	N/A	
Did auditor examine quality data?	YES	NO	N/A	
Did auditor review documents?	YES	NO	N/A	
Did auditor study quality records?	YES	NO	N/A	
Did auditor examine manuals?	YES	NO	N/A	
Did auditor review work activities?	YES	NO	N/A	
Did auditor evaluate processes?	YES	NO	N/A	
Did auditor answer all checklist questions?	YES	NO	N/A	
Did auditor document observations?	YES	NO	N/A	

Did auditor document evidence?	YES	NO	N/A	
Did auditor document conclusions?	YES	NO	N/A	
Did auditor document nonconformities?	YES	NO	N/A	
Did auditor stay on track within the scope of the audit?	YES	NO	N/A	
Did auditor make fair evaluations?	YES	NO	N/A	
Did auditor make realistic judgments?	YES	NO	N/A	
Did auditor make objective assessments?	YES	NO	N/A	
Did auditor make reasonable observations?	YES	NO	N/A	
Did auditor behave in a professional manner?	YES	NO	N/A	
Did auditor treat auditee personnel with courtesy?	YES	NO	N/A	
Did auditor respect and protect the confidentiality of all auditee documents, records, and manuals?	YES	NO	N/A	
Did auditor have the necessary authority?	YES	NO	N/A	
Did auditor have the necessary resources?	YES	NO	N/A	
Were auditor(s) given enough time to do the job properly?	YES	NO	N/A	
Did auditor prepare a detailed Audit Plan?	YES	NO	N/A	
Did auditor prepare a proper Audit Summary?	YES	NO	N/A	
Did auditor prepare a Audit Report?	YES	NO	N/A	
Did auditor organize an effective internal audit?	YES	NO	N/A	
Have auditee managers agreed to a follow-up audit?	YES	NO	N/A	
Does the Internal Audit Report identify nonconformities?	YES	NO	N/A	
Does the Internal Audit Report summarize audit evidence?	YES	NO	N/A	
Does Internal Audit Report discuss audit conclusions?	YES	NO	N/A	

Does the Internal Audit Report recommend corrective actions?	YES	NO	N/A	
Does the Internal Audit Report recommend preventive actions?	YES	NO	N/A	
Does the Audit Report review and evaluate how well the quality policy is being applied?	YES	NO	N/A	
Does the Audit Report review and evaluate how well quality procedures are being followed?	YES	NO	N/A	
Does the Audit Report review and evaluate how well quality plans are being followed?	YES	NO	N/A	
Does the Audit Report review and evaluate how well quality objectives are being achieved?	YES	NO	N/A	
Does the Audit Report review and evaluate the overall performance of system?	YES	NO	N/A	
Does the Audit Report make recommendations about how quality process performance can be improved?	YES	NO	N/A	
Does the Audit Report make recommendations about how quality system performance can be improved?	YES	NO	N/A	
Are you personally satisfied that all important quality nonconformities have been identified?	YES	NO	N/A	
Do you believe that this quality system audit was carried out in a fair and reasonable manner?	YES	NO	N/A	
Do you believe that the Internal Audit Report presents a fair and accurate assessment of the quality system?	YES	NO	N/A	

8.0 References

8.2 A2LA, *ISO/IEC 17025 Assessor Checklist*, August 28, 2000

8.2 A2LA, *ISO/IEC Guide 25 & ISO/IEC 17025 Gaps Assessor Checklist*, August 28, 2000

8.3 ACOE, *Environmental Quality – Validation of Analytical Laboratories*, EM-200-1-1, July 1, 1994.

CT Laboratories Internal Audit
Date of Audit:
Assessor (Initials):

	Data Integrity Assessment	***Document COMPLIANT***			***Assessment COMPLIANT***			**Reference**	**Comments and Corrective Actions**
	Based on: Best Practices for the Detection and Deterrence of Laboratory Fraud	Y	N	na	Y	N	na		
	Ethics Training								
	Does employee have a current (initial and annual) signed copy of the Ethics Agreement on file?								
	Can the interviewed employee cite examples of improper, unethical and illegal actions? Examples could include: Improper use of manual integration to meet QC requirement, intentional manipulation of date and time to meet hold times, falsification of data to meet method requirements, dry-labbing, selective inclusion of data to meet QC, unwarranted manipulation of computer software, improper alteration of analytical conditions, reporting the results of one sample for another, or not performing analytical method as specified in the SOP.								
	Historical								
	If a manual external data validation was performed was the results available identifying non-compliant work? If so, note corrective actions.								
	If an external electronic data audit was performed were the results of problems available to the laboratory? If so, note corrective actions.								
	What corrective action taken as a result of non-compliance or problems identified in the electronic audit?								
	Internal Electronic Data Audit								
	Electronic data checkers (ADR & EQUIS) were errors noted.								
	Instrument software with Audit Trail features, were changes noted in the review.								
	If changes were made to electronic files is both the before and after data available?								

CT Laboratories Internal Audit
Date of Audit:
Assessor (Initials):

	Data Integrity Assessment	***Document COMPLIANT***			***Assessment COMPLIANT***			**Reference**	**Comments and Corrective Actions**
	Based on: Best Practices for the Detection and Deterrence of Laboratory Fraud	Y	N	na	Y	N	na		
	If changes were made to electronic files is the person making the changes identified, date and reason documented?								
	If changes were made were they technically sound?								
	Are Audit Trail files archived?								
	Do file dates match the export dates?								
	Internal Non-electronic Data Audit								
	Instruments without Audit Trail feature were changes noted in the review.								
	If changes were made is both the before and after data available?								
	If changes were made is the person making the changes identified, date and reason documented?								
	If changes were made were they technically sound?								
	Are changes archived?								
	Manual calculations (if present) verified & acceptable?								
	Manual transfer of data to LIMS true & accurate?								
	Has data been excluded?								

Data Validation Checklist

LIMS #:	Method: Anions by Ion Chromatography 300.0/9056			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Were the samples analyzed within hold time?	28 days for Cl, F, SO_4;14 days for preserved NO_2+NO_3; 48 hours for O.Phos, and unpreserved NO_2 and NO_3					
2. Was the calibration performed using the required number of standards?	Three plus a blank					
3. Is the standard prep log number noted on the analytical report?	---					
4. Was the correlation coefficient acceptable?	>= 0.995					
5. Was Calibration Check standard run prior to analysis of samples?	---					
6. Was the Calibration Check standard recovery acceptable?	90 – 110 %					
7. Were the ICV and ICB run immediately after the calibration check standard?	---					
8. Was the ICV recovery acceptable?	90 – 110 %					
9. Was the ICB result acceptable?	< LOD					
11. Were the CCV's and the CCB's analyzed at the required frequency?	1per 10 samples					
12. Were the CCV recoveries acceptable?	90 – 110 %					
13. Were the CCB results acceptable?	< LOD					
14. Was a LCS run at the required frequency?	1 per 20 samples					
15. Was the LCS recovery acceptable?	90-110 %					

16. Was the LCS used before the indicated expiration date?	---					
17. Were the MS and MSD prepared at the required frequency?	1per 20 field samples					
18. Were the MS and MSD recoveries acceptable?	Within in house QC limits					
Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
19. Was the RPD between the MS and MSD acceptable?	Within in house QC limits					
20. Were all chromatograms checked to ensure accurate peak identification?	---					
21. Are all samples on the job lists accounted for?	---					

Data Validation Checklist

LIMS #:	Method: Cyanide			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a distillation sheet accompany the data package?	---					
2. Does the distillation sheet include all reagent and standard prep log numbers?	---					
3. Is a copy of the stock standard standardization log included?	---					
4. Were samples checked for chlorine and sulfide?	---					
5. Were the samples distilled within hold time?	14 days					
6. Was a method blank prepared at the required frequency?	1per 20 samples					
7. Was the method blank result acceptable?	< LOD					
8. Was a LCS prepared at the required frequency?	1per 20 samples					
9. Was the LCS used before the indicated expiration date?	---					
10. Was the LCS recovery acceptable?	Within in house, project, or LCG/ QSM QC limits					
11 Was the MS and MSD prepared at the required frequency?	1per 20 samples (1 per 10 for QSM) per matrix					

Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
12. Were the MS and MSD recoveries acceptable?	Within in house, project, or QSM QC limits					
13. Was the RPD between the MS and MSD acceptable?	Within in house QC limits					
14. Was calibration performed using required number of standards	6 plus a blank					
15. Is the standard prep log number noted on the analytical report?	---					
16. Was the correlation coefficient (r) acceptable?	> or = 0.995					
17. Were distilled High/Low standard recoveries acceptable (QSM/LCG)?	85 – 115 % Rec.					
18. Were the ICV and ICB (if required) run immediately after the calibration curve?	---					
19. Was the ICV recovery acceptable?	85-115 % rec. (90-110 % for Drinking water)					
20. Was the ICB result acceptable?	< MDL / RL					
21. Were the CCV's analyzed at the required frequency?	1per 10 samples					
22. Were the CCV recoveries acceptable?	85-115 % rec. (90-110 % for Drinking water)					
23. Was a MB analyzed at the required frequency?	1 per 20 per matrix					
24. Was the MB results acceptable?	< MDL / RL, project specific, or <1/2 RL for LCG / QSM					
25. Was a sample run in Duplicate and was precision acceptable (QSM/LCG data)?	+/- 20 % Diff.					
26. Are all samples on the job lists accounted for?	---					

Table 4
8330 Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.

Requirement:	**Acceptance Criteria**	**Analyst Review**		**Independent Review**		**Comments:** (indicate reference to an attachment if necessary)
		Yes	**No**	**Yes**	**No**	
1. INITIAL CALIBRATION (ICAL)						
a. Was the Explosive initial calibration performed using a minimum of five varying standard concentration levels?	Lowest standard at or near MRL					
b. Is the variation between calibration response factors for all concentration levels <20% RSD, is r^2 >.990, or r > 0.995 for the regression line?	RSD <20%, r^2 >.990, r>0.995					
c. Was each ICAL uniquely identified (i.e. Standard Number)?						
d. Was an initial calibration blank (ICB) analyzed?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Was there a second source ICV for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
c. Were the recoveries for the ICV between 85-115% (80-120% for QSM).	% Recovery					
d. Was the ICV uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run?						
b. Were the recoveries for the CCVs within program limits?	%Recovery					
c. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8330 Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was a method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	Per program					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	Per program					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	Per program					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	Per program QSM: 30%RPD					

Table 4
8330 Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
f. Was there a acetonitrile:H2O injection preformed prior to sample analysis?						
9. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the PDF file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Data Validation Checklist

LIMS #:	Method: Flashpoint EPA Method 1010			
Analysis Date	Analyst / Data Interpretor	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Was LCS within acceptable limits?	77.9-81.9 F					
2. Were duplicates analyzed at the a ppropriate frequency	1 every 20 samples of the same matrix					
3. Were the duplicates within acceptable limits?	Differ by ≤ 5 F					
2. Are all samples on the job lists accounted for?	---					

GFAA / FLAA / CVAA Data Review checklist			Method: 200.9 7000 series AA 245.1 7470/7471		
Instrumentation: PE SIMAA 6000	VARIAN SPECTRAA10		CETAC HG ANALYZER		
Analysis Date:	Data File:		Date Review:		**Analyte:**_____
Cal Std ID:	LIMS # :	Analyst:	Reviewer:		Approved? Yes No
Calibration Parameters -	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV 90-110% 95-105%					
3) ICB ABS of LOD or RL					
6) CRA - (2X CRDL or LOD) 80-120%					
7) CCV1/CCB1- 90-110% / 80-120%					
8) CCV2/CCB2 ABS of LOD or RL					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
12) CCV6/CCB6					
Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	**Comments:**
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					

GFAA / FLAA / CVAA Data Review checklist			Method: 200.9 7000 series AA 245.1 7470/7471		
Instrumentation: PE SIMAA 6000 Varian Spec-20 Varian Spec-10					
Analysis Date:	Data File:		Date Review:		**Analyte:**_____
Cal Std ID:	LIMS # :	Analyst:	Reviewer:		Approved? Yes No

Calibration Parameters -	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV 90-110% 95-105%					
3) ICB ABS of LOD or RL					
6) CRA - (2X CRDL or LOD) 80-120%					
7) CCV1/CCB1- 90-110% / 80-120%					
8) CCV2/CCB2 ABS of LOD or RL					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
12) CCV6/CCB6					

Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	**Comments:**
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					
Prep Batch ID#:_____ Dig. Meth._____					
Prep. Blank - <LOD or RL					
LCS - generated limits;attached list					
Spiked samples in batch:					
a)_____ matrix = _____					
b)_____ matrix = _____					
c)_____ matrix = _____					
d)_____ matrix = _____					
e)_____ matrix = _____					
PDS 85-115% sample#_____					
MSA Performed? Yes No					

Table 4

Analytical Run# **8082 PCB Analysis Data Review Checklist**

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the PCB initial calibration (Aroclor 1016/1260) performed using a minimum of five varying standard concentration levels on two dissimilar columns?	Lowest standard at or near MRL					
b. Is the variation between calibration response factors for all concentration levels <20% RSD, is r^2 >.990, or r > 0.995 for the regression line?	RSD <20%, r^2 >.990, r>0.995					
c. Was each ICAL uniquely identified (i.e. Standard Number)?						
d. Were there Calibration Factors (CF) established for the remaining Aroclors?						
d. Was an initial calibration blank (ICB) analyzed?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there second source ICVs for all Aroclors analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the recoveries for the ICVs within program limits?	%Recovery					
c. Was the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs (Aroclor 1016/1260) analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run? QSM = every ten sample injections.						
b. Were the recoveries for the CCVs within program limits?	%Recovery					
c. Were confirmed Aroclor detects processed using the appropriate Aroclor method?						
d. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8082 PCB Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was the method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch < 20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a . frequency one per Prep Batch?	Batch < 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	In-house limits or client specified limits					

Table 4
8082 PCB Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. acid treatment, florisil, and sulfur treatment)	Acid, Florisil, GPC, Sulfur Treatments					
f. Was there a hexane injection preformed prior to sample analysis?						
g. Was there a priming standard injected prior to sample analysis?						
9. RECORDS AND REPORTING						
a. Is the Analytical Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, analytical data, and method transfer to PDF format?						
b. Are all chromatograms stamped with initials and date?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier in LIMS and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Table 4

Analytical Run# **8081 Pesticide Analysis Data Review Checklist**

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the Pesticide initial calibration performed using a minimum of five varying standard concentration levels on two dissimilar columns?	Lowest standard at or near MRL					
b. Was there initial calibration performed using a minimum of five varying standard concentration levels for Toxaphene and tech-Chlrodane.						
c. Is the variation between calibration response factors for all concentration levels <20% RSD, is r >.995, or r^2 > 0.990 for the regression line?	RSD <20%, r >.995, r^2>0.990					
d. Was each ICAL uniquely identified (i.e. Standard Number)?						
e. Was an initial calibration blank (ICB) analyzed?						
f. Was there an Evaluation standard injected prior to initial calibration?	Breakdown ≤15%					
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the recoveries for the ICVs within program limits?	%Recovery					
c. Was the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run?						
b. Were there a CCV for Toxaphene and tech-Chrodane at the beginning or at the end of . the analytical sequence?						
c. Were the recoveries for the CCVs within program limits?	%Recovery					
d. Was each CCV uniquely identified (i.e. Standard Number)?						

Table 4
8081 Pesticide Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
4. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?	In-house limits or client specified limits					
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Was there a Toxaphene and tech-Chlordane LCS preformed						
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					
7. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a . frequency one per Prep Batch?	Batch ≤20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) or laboratory control spike (LCS) and its' duplicate (LCSD) within the acceptance criteria?	In-house limits or client specified limits					

Table 4
8081 Pesticide Analysis Data Review Checklist Continued

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. florisil, GPC, and sulfur treatment)	Florisil, GPC, and/or Sulfur Treatments					
f. Was there a hexane injection preformed prior to sample analysis?						
g. Was there a priming standard injected prior to sample analysis?						
h. Was there an Eval standard injected prior to sample analysis and every twelve hours?	Breakdown $\leq$15%					
9. RECORDS AND REPORTING						
a. Is the Analytical Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, analytical data, and method transfered to PDF format?						
b. Are all chromatograms stamped with initials and date?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier in LIMS and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Data Validation Checklist

LIMS #:	Method: pH EPA 150.1			
Analysis Date	Analyst / Data Interpretor	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Was the calibration slope recorded on the bench sheet?	---					
2. Were duplicates analyzed at the appropriate frequency?	1 every 20 samples of the same matrix					
3. Were the duplicates within acceptable limits?	Differ by $\leq$ 0.10 pH units					
4. Are all samples on the job lists accounted for?	---					

Data Validation Checklist

LIMS #:	Method: REACTIVE CYANIDE	

Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a distillation sheet accompany the data package?	---					
2. Does the distillation sheet include all reagent and standard prep log numbers?	---					
3. Is a copy of the stock standard standardization log included?	---					
4. Is the stock standard within acceptance limits?	+/- 0.5% of reported value					
5. Were samples checked for chlorine and sulfide?	---					
6. Were the samples distilled within hold time?	14 days					
7. Were method blanks prepared at the required frequency?	1per 20 samples					
8. Was the method blank result acceptable?	< LOD					
9. Were LCS's prepared at the required frequency?	1per 20 samples					
10. Was the LCS used before the indicated expiration date?	---					
11. Was the LCS recovery acceptable?	Program specific or within in-house QC limits					
12. Were the MS and MSD prepared at the required frequency?	1per 20 samples of the same matrix					
13. Were the MS and MSD recoveries acceptable?	Program specific or within in-house QC limits					
14. Was the RPD between the MS and MSD acceptable?	Program specific or within in-house QC limits					

Requirement:	Acceptance	Analyst Review		Independent Review		Comments:
	Criteria	Yes	No	Yes	No	(indicate reference to an attachment if necessary)
15. Was calibration performed using required number of standards	6 plus a blank					
16. Is the standard prep log number noted on the analytical report?	---					
17. Was the correlation coefficient acceptable?	>= 0.995					
18. Were distilled calibration standard recoveries acceptable?	90-110					
19. Were the ICV and ICB run immediately after the calibration curve?	---					
20. Was the ICV recovery acceptable?	90 – 110 %					
21. Was the ICB result acceptable?	< LOD					
22. Were the CCV's and the CCB's analyzed at the required frequency?	1per 10 samples					
23. Were the CCV recoveries acceptable?	90 – 110 %					
24. Were the CCB results acceptable?	< LOD					
25. Were the samples analyzed within 3 days of being distilled?	---					
26. Was a sample run in Duplicate and was precision acceptable (QSM/LCG data)?	+/- 20 % Diff.					
27. Were distilled High/Low standard recoveries acceptable (QSM/LCG)?	85 – 115 % Rec.					
28. Are all samples on the job lists accounted for?	---					

Data Validation Checklist

LIMS #:	Method: CYANIDE SCREENING			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Does a bench sheet accompany the data package?	---					
2. Does the bench sheet include all reagent numbers?	---					
3. Were the samples analyzed within hold time?	14 days					
4. Were method blanks prepared at the required frequency?	1per 20 samples					
5. Was the method blank result acceptable?	Negative					
6. Were LCSs prepared at the required frequency?	1per 20 samples					
7. Was the LCS used before the indicated expiration date?	---					
8. Was the LCS recovery acceptable?	Positive					
9. Was a MS prepared at the required frequency?	1per 20 samples of the same matrix					
10. Was the MS recovery acceptable?	Positive					
11. Are all samples on the job lists accounted for?	---					

Table 5
8270 Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MRL					
b. SPCC responses.	Avg. RRF ≥ 0.05					
c. Linearity.	RSD≤ 15%, ≤ 30% for CCCs, and r ≥ 0.990 for regression.					
d. Were the standards used for the ICAL uniquely identified?						
e. Was there a DFTPP standard analyzed prior to the ICAL?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the SPCC within QC limits	RRF ≥ 0.05					
c. Were the CCCs within QC limits	%D ≤ 20%					
d. Were the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs for target analytes analyzed at the beginning of the sequence and after every 12 hours.						
b. Were SPCC compounds acceptable?	RRF ≥ 0.050					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
c. Were the CCCs compounds acceptable?	%D ≤ 20%					
d. Were the recoveries for the CCVs acceptable?	%D≤20%, 4 analytes ≤40%.					
e. Was each CCV uniquely identified (i.e. Standard Number)?						
4. DFTPP						
a. Was a DFTPP tune check ran at the beginning of every twelve hour shift?						
b. Were the relative abundance criteria met?						
c. Was the peak tailing < 5 on Pentachlorophenol and < 3 on Benzidine?						
d. Was the breakdown of DDT to DDE and DDD less than 20%						
5. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Were the MB results less than the reporting limit (RL)?	< MRL					
If no, were positive hits in the samples < 5x the amount in the blank flagged with a "B".	< 5x (qualify data) > 5x (no action)					
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch < 20 samples					
6. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits					
If no, and the recoveries were low, flag those analytes "Q". If the recoveries were high, only flag the detects (>RL) for those analytes "Q".						
7. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits					
c. Is the relative percent difference (RPD) for each analyte between a matrix spike (MS) and matrix spike duplicate (MSD) within the acceptance criteria? (same criteria for LCS and LCSD)	20% RSD or In-house limits					
9. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. acid treatment, acid-base fractioning, and Florisil treatment)	GPC, Acid/Base, Florisil, Treatments					
10. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the data file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Table 5
8270 SIM Exp. Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MRL					
b. Linearity.	RSD≤ 15%, ≤ 30% for CCCs, r ≥ 0.995 , r2 > 0.990 for regression.					
c. Were the standards used for the ICAL uniquely identified?						
d. Was there a DFTPP standard analyzed prior to the ICAL?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the CCCs within QC limits	%D ≤ 20%					
c. Were the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs for target analytes analyzed at the beginning of the sequence and after every 12 hours.						

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
b. Were the recoveries for the CCVs acceptable?	%D≤20%,					
c. Was each CCV uniquely identified (i.e. Standard Number)?						
4. DFTPP						
a. Was a DFTPP tune check ran at the beginning of every twelve hour shift?						
b. Were the relative abundance criteria met?						
c. Was the peak tailing < 2 on Pentachlorophenol and < 2 on Benzidine?						
d. Was the breakdown of DDT to DDE and DDD less than 20%						
5. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Were the MB results less than the reporting limit (RL)?	< MRL					
If no, were positive hits in the samples flagged with a "B".						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤ 20 samples					
6. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits					
If no, and the recoveries were low, flag those analytes "Q". If the recoveries were high, only flag the detects (>RL) for those analytes "Q".						
7. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits					

Table 5
8270 Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits					
c. Is the relative percent difference (RPD) for each analyte between a matrix spike (MS) and matrix spike duplicate (MSD) within the acceptance criteria? (same criteria for LCS and LCSD)	20% RSD or In-house limits					
9. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extraction?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. GPC, acid-base fractioning, or silica gel treatment)	Acid/Base, GPC, Silica gel					
10. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the data file?						
b. Are all chromatograms dated and initialed?						
c. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
d. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
e. Is the ICAL method referenced on the Raw Data?						

Table 4. Review Checklist

INDEPENDENT DATA REVIEW CHECKLIST			Method: GCMS (EPA SW-846 8260)		
Analysis Date	Analyst/Data Interpreter	Independent Reviewer	Date or Review	Approved?	
				Yes	No
Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.					

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments:
		Yes	No	Yes	No	
I. BFB Tune Check						
BFB tune check analyzed?	Relative abundance criteria met?					
II. Initial Calibration						
Was initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MDL/LOD?					
Were SPCC compounds acceptable?	Avg. RRF> 0.1; 0.3?					
Were CCC RF's less than 15% ? (Or alternatively no greater than 30% if generating results using linear regression?)						
Was Initial Calibration Blank analyzed?						
Were the ICB results for all target analytes less than the limit of detection (LOD).	<LOD or < project/program specific limits					
III. Continuing Calibration Verification (CCV)						
Was a BFB tune check run at the beginning of every twelve hour shift?	Relative abundance criteria met?					
Was a CCV analyzed after every 12 hour tune check?						
Were SPCC compounds acceptable?	Avg. RRF> 0.1; 0.3?					
Were CCC compounds acceptable?	%D < 20%					
Were all other compounds acceptable? If yes, then continue run.	%D < 20%					
If No, did number of failures exceed sporadic marginal failure limits?	<10% failing or see project/program limits					If yes, samples reanalyzed
Were detects for outlying compounds qualified?						

Requirement:	AcceptanceCriteria	AnalystReview		Independent Review		Comments:
		Yes	No	Yes	No	
IV. Blanks						
Was method blank (MB) analyzed prior to analysis of samples?						
Were the MB results for all target analytes less than the limit of detection (LOD)?	*All target analytes <LOD or < project/program specific limits					*see SOP for more detailed criteria
If analytes were detected in the MB with no associated positives in the samples, no further action is needed. If analyte detects are greater than the acceptance criteria with associated positives in the samples, flag associated positives with "B".						
V. Laboratory Control Spike (LCS)						
Was a LCS analyzed at a frequency of one per 20 samples (or program specified frequency)?						
Were the LCS recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in-house or project/program specified limits.					
If no, were detects (and non-detects for low recoveries) qualified?						
Was the LCS a second source to the initial calibration standards?						
VI. Laboratory Control Spike Duplicate (LCSD). This section applies only to runs without MS/MSD						
Was a LCSD analyzed at a frequency of one per 20 samples (or program specified frequency)?						
Were the LCSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in house or project/program specified limits.					
If no, were detects (and non-detects for low recoveries) flagged "Q"?						
Is the relative percent difference (RPD) for each analyte between duplicate samples acceptable?	Default 20%, or see in-house or project/program specified limits.					
If No, were detects in samples flagged "Y"?						
VII. Matrix Spike/Matrix Spike Duplicate (MS/MSD)						
Was a MS and a MSD analyzed at a frequency of one each per 20 samples (or program specified frequency)?						
Were the MS/MSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see in-house or client specified limits.					

Requirement:	Acceptance Criteria	AnalystReview		Independent Review		Comments:
VII. MS/MSD continued						
		Yes	No	Yes	No	
If No, were non-detects for analytes with low MS/MSD recoveries and detects < 1x spiked amount flagged "M"?						
Is the relative percent difference (RPD) for each analyte between duplicate samples acceptable?	Default 20%, or see in-house or project/program specific limits.					
If No, were detects in samples <1x the spiked amount flagged "Y"?						
VIII. Sample Analyses						
Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
Were surrogate recoveries for all samples and QC within acceptance criteria?	Default 70-130%, or see in-house or project/program specified limits.					
If no, was the sample reanalyzed and/or flagged "S"?						
Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?	Target upper middle range of calibration curve.					
Did all samples meet hold time and preservation criteria as defined by method/program.						
Was a sample with detects between LOD and 5x LOD reanalyzed if following a sample with high level detects exceeding calibration range?	Detects are not attributed to carryover					
Were all samples and QC injected within 12 hours of BFB tune check?						
Were internal standard recoveries acceptable relevant to associated CCV?	Response = -50 to +200%; Ret. time = +/- 30 sec.					
IX. Records and Reporting						
Is sequence file / injection log present in the data package?						
Were all data, calculations, and values verified in LIMS upon completion of data capture?						
Were reported results with amounts exceeding acceptance criteria flagged with appropriate qualifier and, if needed, a NCR completed?						
Was the integrity of the sample kept intact? If not, was a NCR completed?						

Analytical Data Verification Checklist	
	Review Assessment Codes
Element Name and Review Aspect	**Acceptable:** All criteria were met and the data is considered usable.
	Provisional: Some criteria were exceeded resulting in data qualifier or there was insufficient information available to assess. Some data is considered usable.
	Unaacceptable: No data available for review or the criteria were exceeded to such an extent that the associated data is considedred unusable.
	Not Applicable: This item was not reviewed or does not apply to this data package.
Review	
Completed by	
Title	Project Chemist
Company	Earth Resources Technology, Inc.
Date	
Project Description	
Client	USACE
Site	**Savanna Army Depot Activity, Site 50**
Location	Savanna, IL
Phase	
Contract #	
Project #	
Event Description	
Date(s)	
Sampling Locations	
Sample Matrix	
Phase	
Laboratory Details	
Laboratory Name	CT Laboratories
Location	Baraboo, WI
Additional laboratory(s)	
Laboratory Report Number	
Data files	

Element Name and Review Aspect	**Acceptable:** All criteria were met and the data is considered usable. **Provisional:** Some criteria were exceeded resulting in data qualifier or there was insufficient information available to assess. Some data is considered usable. **Unaacceptable:** No data available for review or the criteria were exceeded to such an extent that the associated data is considedred unusable. **Not Applicable:** This item was not reviewed or does not apply to this data package.				
Analytical Data Package - General	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Case narrative					
Internal laboratory chain of custody					
Sample condition upon receipt and storage records					
Sample chronology (time of receipt, extraction, and					
Identification of QC samples					
Copies of laboratory logs					
Definitions of laboratory qualifiers					
Documentation of individual QC results					
Documentation of laboratory method deviations					
Electronic data deliverables					
QC sample raw data					
QC summary report					
Raw data					
Sample summary report					
Signatures (lab QA manager)					
Analytical Data Package - SW846 Method 8260B	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
Duplicates					
MRL Check					
Laboratory control samples (LCS)					
Matrix spikes/matrix spike duplicates (MS/MSD)					
Method blanks (MB)					
Surrogates					

Element Name and Review Aspect	**Acceptable:** All criteria were met and the data is considered usable. **Provisional:** Some criteria were exceeded resulting in data qualifier or there was insufficient information available to assess. Some data is considered usable. **Unaacceptable:** No data available for review or the criteria were exceeded to such an extent that the associated data is considedred unusable. **Not Applicable:** This item was not reviewed or does not apply to this data package.				
Analytical Data Package - SW846 Method 8330B	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
Duplicates					
MRL Check					
Laboratory blanks					
Laboratory control samples (LCS)					
Matrix spikes/matrix spike duplicates (MS/MSD)					
Surrogates					
Analytical Data Package - SW846 Method 8270	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
Duplicates					
Method blank (MB) reports					
Laboratory control spike (LCS) reports					
Surrogates					
Compound Quantitation					

Element Name and Review Aspect	**Acceptable:** All criteria were met and the data is considered usable. **Provisional:** Some criteria were exceeded resulting in data qualifier or there was insufficient information available to assess. Some data is considered usable. **Unaacceptable:** No data available for review or the criteria were exceeded to such an extent that the associated data is considedred unusable. **Not Applicable:** This item was not reviewed or does not apply to this data package.				
Analytical Data Package - SW846 Method 8081A	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
Duplicates					
Method blank (MB) reports					
Laboratory control spike (LCS) reports					
Surrogates					
Compound Quantitation					
Analytical Data Package - SW846 Method 8082A	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
Duplicates					
Method blank (MB) reports					
Laboratory control spike (LCS) reports					
Surrogates					
Compound Quantitation					
Analytical Data Package - SW846 Method 6010B	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Duplicates					
Laboratory blanks					
Instrument initial calibration reports					
Instrument continuing calibration reports					
MRL Check					
ICP Interference Check					
Matrix spikes/matrix spike duplicates (MS/MSD)					
Serial dilutions					

Element Name and Review Aspect	**Acceptable:** All criteria were met and the data is considered usable.				
	Provisional: Some criteria were exceeded resulting in data qualifier or there was insufficient information available to assess. Some data is considered usable.				
	Unaacceptable: No data available for review or the criteria were exceeded to such an extent that the associated data is considedred unusable.				
	Not Applicable: This item was not reviewed or does not apply to this data package.				
Analytical Data Package - SW846 Inorganic Methods	**Acceptable**	**Provisional**	**Unacceptable**	**Not Applicable**	**Comments/Notes**
Raw data					
Summary data					
Instrument initial calibration reports					
Instrument continuing calibration reports					
MRL Check					
Duplicates					
Laboratory blanks					
Laboratory control samples (LCS)					
Matrix spikes/matrix spike duplicates (MS/MSD)					

METALS 1 - REVIEW OF HOLDING TIMES

INORGANIC ANALYSES

Client: Page_____of_____
Laboratory:
SDG:
Reviewer:
Review Date:
Sample Matrix:

		CLP Metals 6 months for prep and analysis		CLP Mercury 28 days for prep and analysis		CLP Cyanide 14 days for prep and analysis	
Sample Number	Collection Date	prepare/analyze	days	prepare/analyze	days	prepare/analyze	days

** Holding time exceeded*

days = number of days past holding time

[ ] *All holding times were reviewed and found to be in compliance* ***CHECK BOX***

COMMENTS:

METALS 2 - REVIEW OF CALIBRATION

CLP INORGANIC ANALYSES

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix: 0

Date, Time, and Calibration Type	Analyte	Correlation Coefficient ($r \geq 0.995$)	%R	QC Limits	Action Detects/Nondetects	Associated Samples

ICV and CCV QC limits are 90-110% except for Hg, which are 80-120%, and CN, which are 85-115%.

[ ] *All correlation coefficients and %Rs were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *No more than 10 samples were analyzed between successive CCVs -* ***CHECK BOX***

COMMENTS:

METALS 3.a - LABORATORY BLANK REVIEW WORKSHEET

INORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Page_____of_____

	ICBs (Units =)			CCBs (Units =)				PBS mg/kg	PBW µg/L
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Molybdenum									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									

Record all calibration and prep blank values whose concentrations are greater than the IDLs, then select the highest levels for qualification.

[ ] *All associated blanks were reviewed and their concentrations were found to be below the IDL -* ***CHECK BOX***

COMMENTS:

METALS 3.b - LABORATORY BLANK REVIEW

INORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Page_____of_____

Blank ID	Analyte	Conc., units	µg/l Action Level 5x	mg/kg Action Level 5x	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value

[ ] *All associated blanks were reviewed and their concentrations were found to be below the IDL -* ***CHECK BOX***

COMMENTS:

METALS 4 - REVIEW OF ICP INTERFERENCE CHECK SAMPLE RESULTS

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Page_____of_____

Are all ICSAB percent recoveries within the QC limits of 80-120%?
(Yes/No) _____ If not, list below.

Full Sample ID	Analyte	ICSAB %R	Conc./Qualifier

[ ] *All %Rs were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *Spectral interference was not found in the full validation sample(s) -* ***CHECK BOX***

Are the concentrations of Al, Ca, Fe, and Mg in the samples > concentration of their respective levels in the ICS solution? (Yes/No) _____
If so, list highest concentration > IDL found in the ICSA solution which should not be present.

Analyte	Max. ICSA Conc.	Affected Full Sample ID	Sample Conc./Qualifier

In general, qualifications are made if the sample concentration is <10x the ICSA concentration.

METALS 5 - REVIEW OF MATRIX SPIKES

INORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Page_____of_____

Sample ID	Analyte	%R	Sample ID (value/qualifier)	Sample ID (value/qualifier)	Sample ID (value/qualifier)	Sample ID (value/qualifier)	Sample ID (value/qualifier)	Sample ID (value/qualifier)	Sample ID (value/qualifier)

QC Limits = 75-125%, except when the concentration of the QC sample exceeds the amount spiked by a factor of four or more.

** Value outside QC Limits*

[ ] *All Matrix Spike %Rs were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

METALS 6 - REVIEW OF MATRIX DUPLICATES

INORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Sample ID	Analyte	Sample Value	Duplicate Value	RPD	Absolute Difference	Qualifier	Associated Samples

RPD = ± 20% for water samples and ± 35% for soil samples unless either the sample or the duplicate is <5x the CRDL. Instead, the absolute value between sample and duplicate must be < CRDL for water samples and <2x CRDL for soil samples.

[ ] *All %RPDs and absolute differences were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

METALS 7 - REVIEW OF BLANK SPIKES (LCS)

INORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Method	Analyte	%R	QC Limits	Qualifier	Associated Samples

In general, inorganic analyses should have blank spike recoveries of 80-120%.

<u>All</u> inorganic blank spike analytes are subject to qualification.

[ ] *All LCS recoveries were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

METALS 8 - GFAA QUALITY CONTROL

VALIDATION FOR GRAPHITE FURNACE ANALYSES

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix: 0

Analytical spike percent recovery review - list all full sample analytical spike recoveries and any qualifiers that may be necessary.

	Antimony	Arsenic	Lead	Selenium	Thallium	_____
Full Sample ID	%R/Qualifier	%R/Qualifier	%R/Qualifier	%R/Qualifier	%R/Qualifier	%R/Qualifier

Was duplicate precision met for the GFAA injections, including analytical spikes? (Yes/No) _____ If not, list below.

Were MSAs analyzed when appropriate? (Yes/No) _____ If not, list below.

Were MSAs spiked at the correct levels? (Yes/No) _____ If not, list below.

Were MSAs with r<0.995 reanalyzed as appropriate? (Yes/No) _____ If not, list below.

Sample ID	QC limit not met	Lab result	Lab action	Required action	Qualifier

[ ] *All GFAA analytical spike %Rs, duplicate injections, and MSA results were reviewed in the full validation sample(s) and found to be in compliance -* ***CHECK BOX***

Sample ID:

METALS 9 - REVIEW OF ICP SERIAL DILUTIONS

INORGANIC ANALYSES

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix: 0

Sample ID	Analyte	Sample Value	Serial Dilution Result	50x IDL	%D	Associated Samples

Both detects and nondetects are affected if the %D >10% and the concentration of the original sample is >50x the IDL.

[ ] *All serial dilution results were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

METALS 10 - REVIEW OF DETECTION LIMITS

VALIDATION OF INORGANIC REPORT LIMITS

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix: 0

List any RLs that did not agree with RL requirements.

Sample ID	Analyte	Lab RL	Correct RL	Was lab contacted?	If so, result?

Did all sample report limits take into account all appropriate weights, volumes, percent solids, and dilution factors? (Yes/No) _____

List any problems below, noting dates, times, samples affected, and how they were affected. Were the metals IDLs current and less than the CRDL? (Yes/No) _____

Show example calculations for soil report limit quantitation.

[ ] *All reported detection limits for the full validation sample(s) were reviewed and found to be in compliance -* ***CHECK BOX***

Sample ID:

METALS 11 - REVIEW OF FIELD DUPLICATES

CLP INORGANICS ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

Page_____of_____

Method	Analyte	Sample ID	Duplicate ID	Sample Value	Duplicate Value	RPD

Suggested QC guidelines are ± 25% for water samples and ± 50% for soil samples. Data are not qualified on the basis of field duplicate results.

[ ] *Field duplicate precision was reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

METALS 12 - REVIEW OF ANALYTE QUANTITATION

VALIDATION OF INORGANIC SAMPLE RESULTS

Clinet: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix: 0

List any reported results that were not quantitated correctly.

Full Sample ID	Analyte	Lab Result	Correct Result	Was lab contacted?	If so, result?

Did all sample results take into account all appropriate weights, volumes, percent solids, and dilution factors? (Yes/No) _____

List any problems below, noting dates, times, samples affected, and how they were affected. Was the lab contacted? (Yes/No) _____ If so, note the result.

Show example calculations.

[ ] *All reported results were recalculated and verified for the full validation sample(s) -* ***CHECK BOX***

Sample ID:

ORG 1 - REVIEW OF HOLDING TIMES

ORGANIC ANALYSES

Client: Page_____of_____

Laboratory:

SDG:

Reviewer:

Review Date:

Sample Matrix:

	CLP VOA	CLP SVOA	CLP Pesticides/PCBs
	analyze 14 days**	soil: extract 14 days analyze 40 days water: extract 7 days analyze 40 days	soil: extract 14 days analyze 40 days water: extract 7 days analyze 40 days

Sample Number	Collection Date	Analyze	days	Extract	days	Analyze	days	Extract	days	Analyze	days

** Holding time exceeded*

***Holding time for soil samples and water samples with appropriate acid preservation (pH<2). Unpreserved aromatic volatiles in water have a 7-day holding time.*

days = number of days past holding time

[ ] *All holding times were reviewed and are found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 2 - REVIEW OF SURROGATE RECOVERIES

ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

VOLATILES	QC LIMITS Water	QC LIMITS Soil	Sample ID	Sample ID	Sample ID	Sample ID	Sample ID	Sample ID	Sample ID
toluene-d8	88-110	84-138							
bromofluorobenzene	86-115	59-113							
1,2-dichloroethane-d4	76-114	70-121							
			Qualifier	Qualifier	Qualifier	Qualifier	Qualifier	Qualifier	Qualifier
SEMIVOLATILES									
nitrobenzene-d5	35-114	23-120							
2-fluorobiphenyl	43-116	30-115							
terphenyl-d14	33-141	18-137							
phenol-d5	10-110	24-113							
2-fluorophenol	21-100	25-121							
2,4,6-tribromophenol	10-123	19-122							
2-chlorophenol-d4	33-110	20-130							
1,2-dichlorobenzene-d4	16-110	20-130							
			A Qual.	A Qual.	A Qual.	A Qual.	A Qual.	A Qual.	A Qual.
			BN Qual.	BN Qual.	BN Qual.	BN Qual.	BN Qual.	BN Qual.	BN Qual.

PESTICIDE/PCB

			col.1	col.2	col.1	col.2	col.1	col.2	col.1	col.2	col.1	col.2	col.1	col.2	col.1	col.2
tetrachloro-m-xylene	30-150	30-150														
decachlorobiphenyl	30-150	30-150														
			Qualifier		Qualifier		Qualifier		Qualifier		Qualifier		Qualifier		Qualifier	

** Value outside QC Limits*

A = Acid, BN = Base/Neutral

[ ] *All surrogate recoveries were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 3.a - REVIEW OF MATRIX SPIKE/MATRIX SPIKE DUPLICATES

ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

VOLATILES	QC LIMITS (%R/%RPD)		Sample ID			Sample ID			Sample ID		
	Water	Soil	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD
1,1-Dichloroethene	61-145/14	59-172/22									
Trichloroethene	71-120/14	62-137/24									
Benzene	76-127/11	66-142/21									
Toluene	76-125/13	59-139/21									
Chlorobenzene	75-130/13	60-133/21									

SEMIVOLATILES			Sample ID			Sample ID			Sample ID		
	Water	Soil	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD
Phenol	12-110/42	26-90/35									
2-Chlorophenol	27-123/40	25-102/50									
1,4-Dichlorobenzene	36-97/28	28-104/27									
N-nitroso-Di-n-propylamine	41-116/38	41-126/38									
1,2,4-Trichlorobenzene	39-98/28	38-107/23									
4-Chloro-3-methylphenol	23-97/42	26-103/33									
Acenaphthene	46-118/31	31-137/19									
4-Nitrophenol	10-80/50	11-114/50									
2,4-Dinitrotoluene	24-96/38	28-89/47									
Pentachlorophenol	9-103/50	17-109/47									
Pyrene	26-127/31	35-142/36									

PESTICIDE/PCB			Sample ID			Sample ID			Sample ID		
	Water	Soil	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD	MS%R	MSD%R	RPD
gamma-BHC	56-123/15	46-127/50									
Heptachlor	40-131/20	35-130/31									
Aldrin	40-120/22	34-132/43									
Dieldrin	52-126/18	31-134/38									
Endrin	56-121/21	42-139/45									
4,4'-DDT	38-127/27	23-134/50									

** Value outside QC Limits*

[ ] *All MS/MSD recoveries were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 3.b - REVIEW OF BLANK SPIKES (LCS)

CLP ORGANICS ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Method	Analyte	%R	QC Limits	Qualifier	Associated Samples

In general, volatile organic analyses should have blank spike recoveries of 75-125%, and extractable organic analyses should have blank spike recoveries of 60-140%.

[ ] *All LCS recoveries were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 3.c - REVIEW OF MATRIX SPIKE/MATRIX SPIKE DUPLICATES

VOA

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

VOLATILES	QC LIMITS (%R/%RPD) Water	Soil	Sample ID MS%R	MSD%R	RPD	Sample ID MS%R	MSD%R	RPD	Sample ID MS%R	MSD%R	RPD

** Value outside QC Limits*

[ ] *All MS/MSD recoveries were reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 4.a - COMMON BLANK CONTAMINANT REVIEW

ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value
CLP VOA:						
Methylene chloride						
Acetone						
CLP SVOA:						
Dimethylphthalate						
Diethylphthalate						
Di-n-butylphthalate						
Butylbenzylphthalate						
bis(2-Ethylhexyl)phthalate						
Di-n-octylphthalate						

	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value
CLP VOA:						
Methylene chloride						
Acetone						
CLP SVOA:						
Dimethylphthalate						
Diethylphthalate						
Di-n-butylphthalate						
Butylbenzylphthalate						
bis(2-Ethylhexyl)phthalate						
Di-n-octylphthalate						

For qualification of common lab contaminants, use the 5x CRQL rule and the UJb qualifier.

[ ] *All samples were reviewed found to be free of common laboratory contamination -* ***CHECK BOX***

COMMENTS:

ORG 4.b - LABORATORY BLANK REVIEW

ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Page_____of_____

Method, Blank ID	Analyte	Conc., units	Action Level (5x or 10x)	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value	Sample ID old value/new value

[ ] *All associated blanks were reviewed and found to be free of contamination -* ***CHECK BOX***

COMMENTS:

ORG 5 - REVIEW OF GC/MS TUNING AND SYSTEM PERFORMANCE

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

GC/MS TUNING

Was BFB (for VOA) and/or DFTPP (for SVOA) analyzed for each 12-hour period during which samples were analyzed? (Yes/No) _____

Date, time analyzed	BFB or DFTPP?	Base Peak correct?	Abundance Criteria met?	If not, samples affected	Qualification	Comments

SYSTEM PERFORMANCE

Was there any degradation of system performance? (Yes/No) _____

If so, comment below, noting dates, times, samples affected, and how they were affected.

[ ] *All GC/MS tuning and system performance criteria were reviewed and found to be in compliance -* ***CHECK BOX***

ORG 6.a - REVIEW OF CALIBRATION

VOA

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Page_____of_____

Date, Time, and Calibration Type	Analyte	Mean RRF (≥0.05)	%RSD (≤30%)	%D (≤25%)	Action Detects/Nondetects	Associated Samples

[ ] *RRF, %RSD, and %D were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *All calibrations were analyzed at the proper frequency -* ***CHECK BOX***

COMMENTS:

ORG 6.b - REVIEW OF CALIBRATION

SVOA

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Date, Time, and Calibration Type	Analyte	Mean RRF (≥0.05)	%RSD (≤30%)	%D (≤25%)	Action Detects/Nondetects	Associated Samples

[ ] *RRF, %RSD, and %D were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *All calibrations were analyzed at the proper frequency -* ***CHECK BOX***

COMMENTS:

ORG 6.c - REVIEW OF CALIBRATION

PESTICIDE/PCB

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix:

Date, Time, and Calibration Type	Analyte	%Breakdown (≤20% / ≤30%)	%RSD (≤20%)	RPD (≤25%)	Action Detects/Nondetects	Associated Samples

For each PEM in each column, the percent breakdown for 4,4'-DDT and endrin should be ≤ 20% and ≤ 30% combined.

Single component pesticides and surrogates should have a %RSD ≤ 20% except for the surrogates, which should have a %RSD ≤ 30%.

Up to two single component pesticides may have a %RSD >20% but ≤ 30%.

[ ] *Resolution check mixture met resolution criteria of ≥ 60% -* ***CHECK BOX***

[ ] *%Breakdown, %RSD, and RPD were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *All calibrations were analyzed at the proper frequency -* ***CHECK BOX***

COMMENTS:

ORG 7.a - INTERNAL STANDARD REVIEW

VOA

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Method	Sample ID	Internal Std. Name	Area	Standard Area	QC Limit Low	QC Limit High	Qualifier

See worksheet A.8.b for associated compounds.

[ ] *All internal standard area counts were reviewed and found to be in compliance -* ***CHECK BOX***

[ ] *All retention times met specified QC limits -* ***CHECK BOX***

COMMENTS:

ORG 8.b - CORRESPONDING TARGET INTERNAL STANDARD COMPOUNDS

VOA & SVOA

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix:

CLP VOA:

Bromochloromethane	1,4-Difluorobenzene
Chloromethane	1,1,1-Trichloroethane
Bromomethane	Carbon tetrachloride
Vinyl chloride	Bromodichloromethane
Chloroethane	1,2-Dichloropropane
Methylene chloride	trans-1,3-Dichloropropene
Acetone	Trichloroethene
Carbon disulfide	Dibromochloromethane
1,1-Dichloroethene	1,1,2-Trichloroethane
1,1-Dichloroethane	Benzene
1,2-Dichloeoethene (tot.)	cis-1,3-Dichloropropene
Chloroform	Bromoform
1,2-Dichloroethane	
2-Butanone	

CLP SVOA:

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octylphthalate
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-nitroso-diphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenylphenylether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene	Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	2-Nitroaniline	Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethylphthalate	Carbazole	Chrysene	Benzo(g,h,i)perylene
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene	Phenanthrene		Dibenzo(a,h)anthracene
2,2'-oxybis(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene		
4-Methylphenol	4-Chloroaniline	Acenaphthene	Di-n-butylphthalate		
N-nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol			
	2-Methylnaphthalene	Dibenzofuran			
		2,4-Dinitrotoluene			
		2,6-Dinitrotoluene			
		Diethylphthalate			
		4-Chlorophenyl-phenylether			
		Fluorene			
		4-Nitroaniline			

ORG-8 REVIEW OF COMPOUND IDENTIFICATION

GC ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Sample ID	Analysis	Compound	Concentration, Column 1	Concentration, Column 2	%D	Qualifier

[ ] *All %Ds between the columns for detected compounds were reviewed and found to be <50% -* ***CHECK BOX***

COMMENTS:

ORG 9 - REVIEW OF FIELD DUPLICATES

ORGANICS ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Page_____of_____

Method	Analyte	Sample ID	Duplicate ID	Sample Value	Duplicate Value	RPD

Suggested QC guidelines are ± 25% for water samples and ± 50% for soil samples. Data shall not be qualified on the basis of field duplicate results.

[ ] *Field duplicate precision was reviewed and found to be in compliance -* ***CHECK BOX***

COMMENTS:

ORG 10 - REVIEW OF TCL IDENTIFICATION

VALIDATION FOR ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

List any reported compounds that were not within ± 0.06 RRT of the standard RRT.

Sample ID	Compound	Compound RRT	Standard RRT	Was lab contacted?	If so, result?

Were there any other problems with TCL identification such as relative intensities of ions not within ± 20% of the standard spectra? (Yes/No) _____

If so, comment below,noting dates, times, samples affected, and how they were affected. Was the lab contacted? (Yes/No) _____ If so, note the result.

[ ] *All RRTs, relative intensities, and spectra were reviewed for the full validation sample(s) and found to be in compliance -* ***CHECK BOX***

Sample ID:

ORG 11 - REVIEW OF TENTATIVELY IDENTIFIED COMPOUNDS

VALIDATION FOR ORGANIC ANALYSES

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

Were all of the identified TICs quantitated and identified correctly? (Yes/No) _____ List any changes made to the Form 1s, including additions of "NJ"s, if any.

Sample ID	Compound	Change/Addition

Did all TIC results take into account the appropriate weights, volumes, percent solids, and dilution factors? (Yes/No) _____

List any problems below, noting dates, times, samples affected, and how they were affected. Was the lab contacted? (Yes/No) _____ If so, note the result.

Show an example calculation.

[ ] *All of the full validation sample(s) TICs were reviewed and found to be correctly identified and quantitated -* ***CHECK BOX***

Sample ID:

ORG 12 - REVIEW OF ANALYTE QUANTITATION

VALIDATION OF ORGANIC SAMPLE RESULTS

Client: 0

Laboratory: 0

SDG:

Reviewer: 0

Review Date:

Sample Matrix:

List any reported results that were not quantitated correctly.

Sample ID	Analyte	Lab Result	Correct Result	Was lab contacted?	If so, result?

Did all sample results take into account all appropriate weights, volumes, percent solids, and dilution factors? (Yes/No) _____

List any problems below, noting dates, times, samples affected, and how they were affected. Was the lab contacted? (Yes/No) _____ If so, note the result.

Show example calculations.

[ ] *All reported results were recalculated and verified for the full validation sample(s) -* ***CHECK BOX***

Sample ID:

ORG 13 - REVIEW OF DETECTION LIMITS

VALIDATION OF ORGANIC REPORT LIMITS

Client: 0
Laboratory: 0
SDG:
Reviewer: 0
Review Date:
Sample Matrix:

List any RLs that did not agree with RL requirements.

Sample ID	Analyte	Lab RL	Correct RL	Was lab contacted?	If so, result?

Did all sample report limits take into account all appropriate weights, volumes, percent solids, and dilution factors? (Yes/No) _____

Please list any problems below, noting dates, times, samples affected, and how they were affected.

Show example calculations for soil report limit quantitation.

[ ] *All reported detection limits for the full validation sample(s) were reviewed and found to be in compliance -* ***CHECK BOX***

Sample ID:

APPENDIX F
RESUMES