REGION 5 RAC2

REMEDIAL ACTION CONTRACT FOR

Remedial, Enforcement Oversight, and
Non-Time Critical Removal Activities at Sites of Release
or Threatened Release of Hazardous Substances in Region 5

FINAL UNIFORM FEDERAL POLICY
QUALITY ASSURANCE PROJECT PLAN

Southeast Rockford Groundwater Contamination Superfund Site
Technical Assistance
Winnebago County, Illinois

WA No. 175-TATA-05DK/Contract No. EP S5-06-01

January 2014

PREPARED FOR

U.S. Environmental Protection Agency

PREPARED BY

CH2M HILL
Critigen, LLC

FOR OFFICIAL USE ONLY

Final Uniform Federal Policy
Quality Assurance Project Plan

Southeast Rockford Groundwater Contamination Superfund Site Winnebago County, Illinois

Technical Assistance

WA No. 175-TATA-05DK/Contract No. EP-S5-06-01

Prepared for

January 2014

CH2MHILL®

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Acronyms and Abbreviations

AS/SVE	air sparge and soil vapor extraction system
bgs	below ground surface
CC	continuing calibration
CC	calibration check compounds
CCV	continuing calibration verification
CDM	Camp, Dresser & McKee
CLP	Contract Laboratory Program
CMA	Coastal Monitoring Associates
COC	contaminant of concern
CPR	cardiopulmonary resuscitation
CRL	Chicago Regional Laboratory
C/T	specific conductance and temperature
CVAA	cold vapor atomic adsorption
DQI	data quality indicator
DPT	direct-push technology
DQO	data quality objective
EB	equipment blank
EDD	electronic data deliverable
FB	field blank
FD	field duplicate
FOP	field operating procedure
GC/ECD	gas chromatograph/electron capture detector
GC/MS	gas chromatograph/mass spectrometry
GIS	geographic information system
GPS	global positioning system
IC	initial calibration
ICAP	inductively coupled argon plasma
ICP-MS	inductively coupled plasma-mass spectrometry
ICS	Interference check solutions
ICV	initial calibration verification
IDW	investigation-derived waste
IEPA	Illinois Environmental Protection Agency
LCS	laboratory control sample

LNAPL	light nonaqueous phase liquid
MB	method blank
MDL	method detection limit
$\mu g/m^3$	microgram per meter cubed
µg/L	microgram per liter
mg/L	milligram per liter
MIP	membrane interface probe
MNA	monitored natural attenuation
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
ORP	oxidation-reduction potential
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PCB	polychlorinated biphenyl
PCE	tetrachloroethene
PDS	post-digestion spike
%R	percent recovery
PID	photoionization detector
%R	percent recovery
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RF	response factor
RI	remedial investigation
RL	reporting limit
ROD	record of decision
RPD	relative percent difference
RRT	relative retention time
RSD	relative standard deviation
RSL	Regional Screening Level
SOP	standard operating procedure
SPCC	system performance check compounds

SSC	site safety coordinator
SVOC	semivolatile organic compound
TBD	to be determined
TCA	1,1,1-trichloroethane
TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
TDS	total dissolved solids
USEPA	U.S. Environmental Protection Agency
UST	underground storage tank
VISL	Vapor Intrusion Screening Level
VOC	volatile organic compound

Worksheets #1 and #2—Title and Approval Page

Site Name/Project Name: Southeast Rockford Groundwater Contamination Superfund Site

Revision Number: 0

Site Location: Winnebago County, Illinois **Site Number/Code:** ILD981000417

Operable Unit: NA

Document Title: *Quality Assurance Project Plan: Southeast Rockford Groundwater Contamination Superfund Site*

Lead Organization: U.S. Environmental Protection Agency (USEPA)

Contractor Name: CH2M HILL **Contractor Number:** EP-S5-06-01

Contract Title: Technical Assistance **Work Assignment Number:** 175-TATA-05DK

Preparer's Name and Organizational Affiliation: Shannon Olson/CH2M HILL

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Preparation Date (Day/Month/Year): 11/01/2013

1. Identify regulatory program:

   The Comprehensive Environmental Response, Compensation, and Liability Act of 1980, commonly known as Superfund

2. Identify approval entity: USEPA Region 5
3. The Quality Assurance Project Plan (QAPP) is (select one): ☐Generic ☒Project-Specific
4. List dates of scoping sessions that were held: August 28, 2013
5. List dates and titles of QAPP documents written for previous site work, if applicable: NA
6. List organizational partners (stakeholders) and connection with lead organization:

   USEPA Region 5 (lead organization)
   CH2M HILL (contractor)

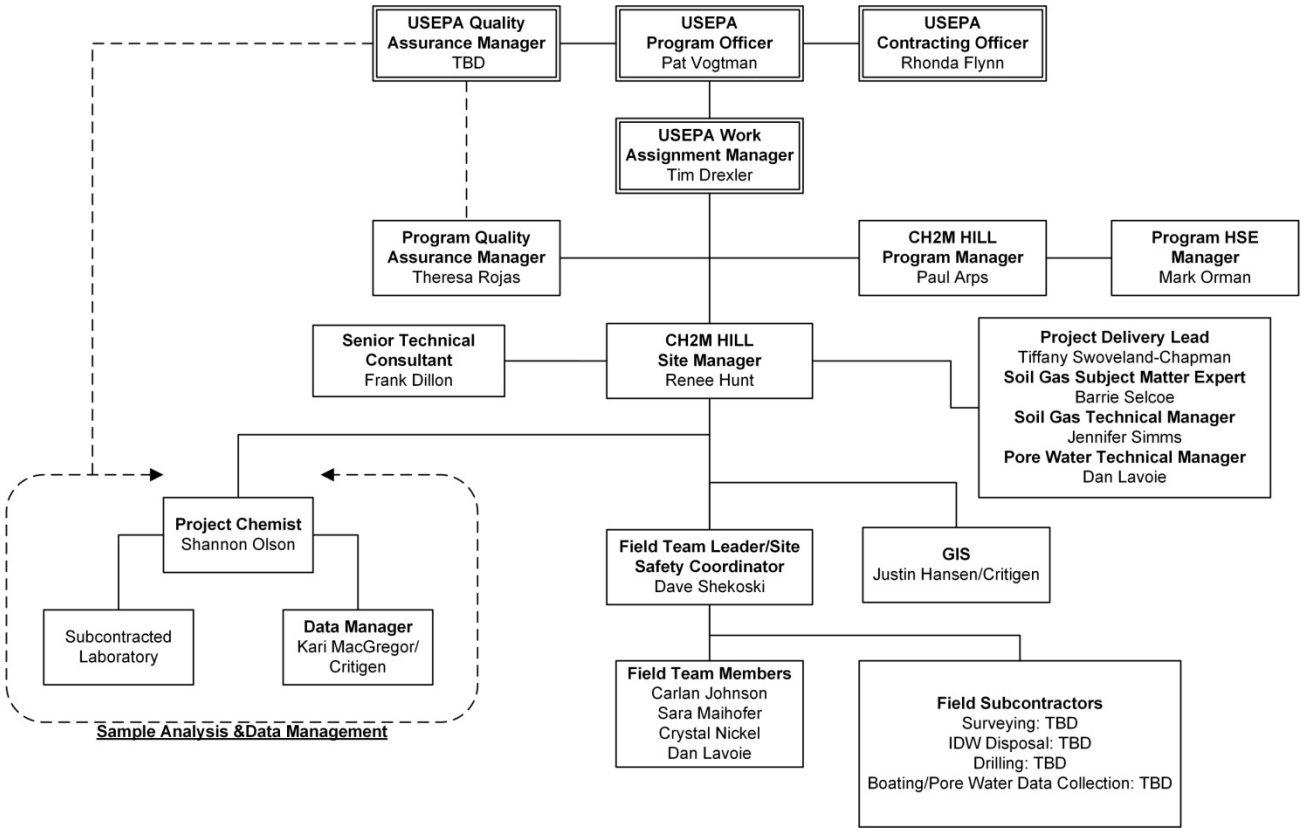
7. List data users:

   USEPA, CH2M HILL, Illinois EPA (IEPA)

Investigative Organization's Site Manager	1/17/14 [Date]	Renee M. Hunt Renee Hunt/CH2M HILL
Investigative Organization's Site Quality Assurance (QA) Officer	1/17/14 [Date]	Theresa Rojas Theresa Rojas/CH2M HILL
Investigative Organization's Project Chemist	1/17/14 [Date]	Shannon M. Olson Shannon Olson/CH2M HILL
Lead Organization's Project Manager	1/15/14 [Date]	Timothy Drexler/USEPA
Region 5 Project Chemist	1/15/14 [Date]	Michael McDonough Michael McDonough/USEPA
Quality Assurance Approval Authority	1/16/14 [Date]	Michael McDonough USEPA Region 5
Laboratory Director	1/16/14 [Date]	Michael J. Healy TestAmerica/Laboratory Director Michael J. Healy
Laboratory QA Manager	1/15/14 [Date]	Terese A. Preston TestAmerica/Laboratory QA Manager Terese Preston

Document Control Numbering System: SR-TA-R0

Worksheets #3 and #5—Project Organization and QAPP Distribution



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Dave Shekoski	Field team leader and site safety coordinator (SSC)	CH2M HILL	414-847-0345	414-272-4408	Dave.Shekoski@ch2m.com	SR-TA-R0-12
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Kari MacGregor	Database manager	Critigen	262-377-5594	720-872-4129	Kari.Macgregor@critigen.com	SR-TA-R0-15
Justin Hansen	Geographic information system (GIS) manager	Critigen	414-847-0244	414-454-8830	Justin.Hansen@critigen.com	SR-TA-R0-16

Worksheets #4, #7, and #8—Personnel Qualifications and Signoff Sheet

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Timothy Drexler	Region 5 work assignment manager	USEPA	Overall responsibility for all phases of work, review, and approval	
Michael McDonough	Region 5 chemist	USEPA	QAPP review and approval	
Paul Arps	Program manager	CH2M HILL	Overall responsibility for meeting USEPA objectives and CH2M HILL quality standards, as well as technical quality control (QC) and project oversight; QAPP review	B.S., Chemistry, 13 years of experience
Theresa Rojas	QA manager	CH2M HILL	QA review	B.S., Chemistry, 25 years of experience
Renee Hunt	Site manager	CH2M HILL	Administrative, decision, and approval authority	M.S., Environmental Engineering, B.S., Civil Engineering, 12 years of experience
Frank Dillon	Senior technical consultant	CH2M HILL	Provides guidance to the site manager on managing the review team	M.S., Biology, B.A., Zoology, 29 years of experience
Tiffany Swoveland Chapman	Project delivery lead	CH2M HILL	Provides assistance to the site manager on all deliverables	B.S., Geological Engineering, 13 years of experience
Daniel Lavoie	Pore water technical manager	CH2M HILL	Provides technical pore water expertise and develops site-specific goals for the site	M.S., Biology/Aquatic Ecotoxicology, B.S., Biology/Environmental Science, 12 years of experience
Barrie Selcoe	Soil gas subject matter expert	CH2M HILL	Provides technical vapor intrusion expertise and develops site-specific goals for the site	M.P.H., Risk Assessment, B.S., Environmental Health, 25 years of experience
Jennifer Simms	Soil gas technical manager	CH2M HILL	Conducts vapor intrusion assessments and develops site-specific goals	B.S., Environmental Engineering, 10 years of experience
Mark Orman	Health and safety operations manager	CH2M HILL	Conducts health and safety oversight	B.S., Environmental Studies, 16 years of experience
Dave Shekoski	Field team leader	CH2M HILL	Coordinates, directs, participates in, and reports site activities; ensures adherence to the QAPP, and communicates issues to site manager and field team; contractor oversight	A.S., Applied Sciences, 26 years of experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Dave Shekoski	SSC	CH2M HILL	Oversees health and safety for all field activities	A.S., Applied Sciences, 26 years of experience
Shannon Olson	Project chemist	CH2M HILL	Assists in QAPP preparation, coordinates laboratory subcontractors, and performs oversight of laboratory and data validation, performs data evaluation	M.S., Environmental Engineering, B.S., Environmental Science, 6 years of experience
Kari MacGregor	Data manager	Critigen	Performs management of team subcontract and provides senior technical support for data management activities	TBD
Justin Hansen	GIS manager	Critigen	Provides GIS support	TBD
Jim Knapp	Laboratory program manager	TestAmerica	Manages sample tracking and maintains communication with the project chemist	TBD

Special Training Requirements

Project Function	Specialized Training Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities	Hazardous waste operations 40-hour training; 8-hour refresher	Registered training organization	Annually	All field staff	Field team staff	CH2M HILL Human Resources Department
Field Activities	Cardiopulmonary resuscitation (CPR) and first-aid	Registered training organization	Every 3 years	All field staff	Field team staff	CH2M HILL Human Resources Department
Field Activities	SSC–Hazardous Waste	Registered training organization	Every 3 years	SSC	SSC from CH2M HILL	CH2M HILL Human Resources Department
Health and Safety	Health and safety plan	CH2M HILL	Various	All field personnel and subcontracted project personnel working in the field	All field personnel from CH2M HILL and subcontracted personnel	Signoff sheet at the end of the health and safety plan

Signoff Sheet

Organization: USEPA

Project Personnel	Title	Telephone Number	Signature	Date
Timothy Drexler	Region 5 work assignment manager	312-353-4367		1/15/14
Michael McDonough	Region 5 Chemist	312-886-1550	Michael McDonough	1/15/14

Organization: CH2M HILL

Project Personnel	Title	Telephone Number	Signature	Date
Paul Arps	Program manager	262-349-4180		1/17/14
Theresa Rojas	QA manager	678-530-4297		1/17/14
Renee Hunt	Site manager	414-847-0349	Renee M. Hunt	1/17/14
Frank Dillon	Senior technical consultant	517-996-6034		11/01/2013
Tiffany Swoveland Chapman	Project delivery lead	314-335-3044		1/17/14
Shannon Olson	Project chemist	503-736-4111	Shannon M. Olson	11/01/2013

Worksheet #6—Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Communication with CH2M HILL program manager	USEPA program officer	Pat Vogtman	312-886-9553	Provides administrative direction to CH2M HILL program manager and project team, authorizes changes to plan and can stop work, if needed.
Communication with CH2M HILL contract manager	USEPA Contracting Officer	Rhonda Flynn	312-353-3187	Provides administrative direction to CH2M HILL contracts manager.
Communication with USEPA program officer and Contracting Officer	CH2M HILL program manager	Paul Arps	262-349-4180	Receives contractual direction from USEPA program officer and contracting officer, and notifies USEPA of contractual deviations (changes in scope of work, budget, or schedule) by e-mail or letter.
Communication with CH2M HILL site manager	USEPA work assignment manager	Timothy Drexler	312-353-4367	Serves as primary point of contact for USEPA, and provides approval of technical direction to CH2M HILL site manager.
Point of contact with USEPA work assignment manager	CH2M HILL site manager	Renee Hunt	414-847-0349	All materials and information about the project will be forwarded to Timothy Drexler by Renee Hunt.
Field staff discussion and inquiry	CH2M HILL field team leaders	Dave Shekoski	414-847-0345	Serve as a primary point of contact for field team before, during, and after the investigation; communicates back to the site manager, quality manager, and project chemist, as needed. Communication by phone as needed with field staff during field sampling events, followed up with e-mail to document decisions and actions.
Health and safety	CH2M HILL SSC(s)	Dave Shekoski	414-847-0345	Responsible for the adherence of team members to the site safety requirements described in the health and safety plan. Will report health and safety incidents and near misses to the site manager and health and safety operations manager.
QAPP changes in the field	CH2M HILL field team leaders	Dave Shekoski	414-847-0345	The field team leader will notify the site manager by phone and e-mail of changes to the QAPP made in the field and the reasons within 24 hours. Documentation of deviations from the work plan will be kept in the field logbook; deviations made only with the approval of the contractor site manager. The site manager will advise Timothy Drexler of all changes.
Daily field progress reports	CH2M HILL field team leaders	Dave Shekoski	414-847-0345	E-mail or fax daily field progress reports to Renee Hunt.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Field corrective actions	CH2M HILL field team leaders	Dave Shekoski	414-847-0345	The need for corrective action for field issues will be determined by the field team leader. The site manager will ensure QAPP requirements are met by field staff. The field team leader will notify the site manager of any needed field corrective actions. The site manager will have 24 hours to respond to the request for field corrective action.
Reporting laboratory data quality issues	TestAmerica, Inc., Laboratory	Jim Knapp	708-534-5200	All QA/QC issues with field samples will be reported to the project chemist immediately.
Analytical corrective actions	Project chemist	Shannon Olson	503-736-4111	The need for corrective action by the analytical laboratory will be determined by the project chemist. The project chemist will ensure QAPP requirements are met by the laboratory. No analytical data can be released until data is reviewed for completeness and conformance to analytical guidelines by the project chemist. The project chemist will review all data as soon as possible upon receipt from the validator.
Release of analytical data	TestAmerica, Inc., Laboratory	Jim Knapp	708-534-5200	No analytical data can be released to CH2M HILL and USEPA until it has been reviewed by the laboratory. No final data can be released to CH2M HILL until validation is completed and the laboratory has approved the release.
QAPP amendments	Region 5 work assignment manager and chemist	Timothy Drexler, Alida Roberman	312-353-4367 847-256-3237	Any major changes to the QAPP must be approved before the changes can be implemented.

Worksheet #9—Project Planning Session Summary

Site Name:	Southeast Rockford Groundwater Contamination Superfund Site
Project Name and Site Location:	Technical Assistance—Southeast Rockford Groundwater Contamination Superfund Site, Winnebago County, Illinois
Projected Date(s) of Sampling:	November 2013 through February 2014
Site Manager:	Renee Hunt

Date of Session: August 28,2013

Scoping Session Purpose: Discuss use of historical data in selecting soil gas probe sampling locations. Discuss the limitations of the data and information needed to perform the soil gas sampling.

Name	Title/Role	Affiliation	Phone #	E-mail Address
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Keith Fusinski	USEPA risk assessor	USEPA	734-692-7681	fusinski.keith@epa.gov
Renee Hunt	Site manager	CH2M HILL	414-847-0349	Renee.Hunt@ch2m.com
Frank Dillon	Senior technical consultant	CH2M HILL	517-996-6034	Frank.Dillion@ch2m.com
Jennifer Simms	Soil gas technical manager	CH2M HILL	215-640-9071	Jennifer.Simms@ch2m.com
Barrie Selcoe	Soil gas subject matter expert	CH2M HILL	281-246-4322	Barrie.Selcoe@ch2m.com

Decisions/Discussion Items

The project team concluded that for soil gas probe placement, the areas of high potential volatile organic compound (VOC) concentrations should be investigated. Tim Drexler indicated that Area 7 is a concern. It was discussed whether there was a remaining vadose zone VOC source at each of the four source areas. USEPA indicated that Area 7 and Area 4 have remaining vadose zone VOC contamination. Much of the VOC source has been removed in Area 9/10 through various remediation efforts. No vadose zone VOC contamination has been identified at Area 11. It was clarified that sample locations will be selected primarily based on current land use and not to focus on potential future receptors. Keith Fusinski indicated that historical soil gas data should be disregarded when determining sample placement.

The depth of soil gas probe placement was also discussed based upon groundwater depth. It was indicated that the annual groundwater monitoring reports may be able to provide information regarding seasonal changes/ variations in groundwater depth over time. Keith and Tim agree that installing the soil gas probes three feet above the groundwater table would be appropriate based upon the historical groundwater data.

Follow-up Task

Tim Drexler will check with Doyle Wilson (IEPA) for information regarding the extent of waste remaining in place at Area 7.

Worksheet #10—Conceptual Site Model

10.1 The Problem to be Addressed by the Project

The purpose of this investigation is to evaluate contaminant concentrations in deep soil gas (immediately above the water table) to identify potential human health impacts within residential and commercial portions of the site due to vapor intrusion and to evaluate contaminant concentrations in pore water within the Rock River to identify potential impacts to ecological receptors.

10.2 Site Description

The site (Figure 1 and 2) is located in the City of Rockford, Winnebago County, Illinois and consists of an approximately 7.5 square mile area. The overall site is defined in the June 2002 Record of Decision (ROD) as the area where groundwater contamination exceeds 10 parts per billion of total chlorinated VOCs (USEPA 2002). Benzene, ethylbenzene, toluene, and xylenes are also identified in the ROD as groundwater contaminants of concern. Much of the groundwater plume underlies an area of mixed residential and commercial properties extending from Sandy Hollow Road to North 23rd Avenue and from Alpine Road west to the Rock River.

There are four primary source areas within the Southeast Rockford groundwater contamination site: Area 4, Area 7, Area 9/10, and Area 11. The location of the source areas are described in the following subsections.

Area 4

Area 4 is located south of Marshall Street and north of Alton Avenue. It is bounded to the west by Marshall Street and to the east by Barrett's Mobile Home Park. H&H Pallets (formerly Swebco Manufacturing, Inc.) and Barrett's Mobile Home Park currently occupy Area 4 (USEPA 2013). The ROD (USEPA 2002) indicated that leaking underground storage tanks (USTs), beneath the parking lot of Swebco Manufacturing were believed to be the source of the contamination in Area 4, however recent investigations indicate that the source of contamination in Area 4 was the result of direct placement of waste at the former loading dock area.

Area 7

Area 7 is located to the northwest of the intersection of Alpine Road and Sandy Hollow Road, and at terminus of Balsam Lane. The Ekberg Park, wooded areas, and an unnamed stream are included in Area 7. Residential properties are located to the east and south of Area 7. Historical aerial photographs and maps indicate that a quarry was once present and illegal dumping/waste disposal activities were conducted in the area (USEPA 2002). Based upon the report *Subsurface Geophysical Investigation Ekberg Pine Manor Park* and the extent of waste excavation in Area 7, solid waste remains in place within the source area (Hutchinson 2007).

Area 9/10

Area 9/10 is bounded by 23rd Avenue to the north and Harrison Avenue to the south, and to the east-west is bounded between 6th and 11th Streets. Several industrial facilities are located within Area 9/10, facilities that are currently in operation include the following: Sundstrand Corporations Plant 1, Paoli Manufacturing, Rockford Products Corporation, and J.L. Clark. Industrial activities have been documented in this area since 1926, when the Rockford Milling Machine and Rockford Tool Companies merged into Sundstrand Machine Tool Company. Other primary facilities which occupied this area, however are no longer in operation, include Mid-states Industrial Company (Rockford Power Machinery), Nylint Corporation, and Rohrbacher Manufacturing (USEPA 2002). Potential sources of contamination have been identified in Area 9/10 including:

- **Sundstrand Plant:** Major source areas identified at the Sundstrand plant include the following: (1) An outdoor drum storage area located on Sundstrand Plant 2 near 9th Street and 23rd Avenue. From 1962 to 1985 various chemicals were stored including tetrachloroethene (PCE), 1,1,1-trichloroethane (TCA), toluene, acetone, and methylene chloride; (2) the loading dock at Sundstrand plant 1 had approximately 14 USTs containing solvents (PCE, TCA), cutting oils, fuel oil and jet fuel (JP4) from 1962 to 1987; and (3) the Waste Recycling Area at Sundstrand's Plant 1. Additionally, up to 40 USTs ranging in size from 500 gallons to 10,000 gallons were present at the Sundstrand Plant 1 at any one time. Many of the USTs may have been removed or abandoned in place.
- **Mid-States Industrial (Rockford Power):** A drum storage area at this facility was identified as a potential source area.
- **Nylint:** Elevated concentrations of TCA detected in soil gas at the west end of the building suggest a potential nearby source.
- **Rockford Products:** Elevated concentrations of VOCs detected in soil gas on 9th Street indicate a possible source area.

Area 11

Area 11 is located at the corner of Eleventh Street and Harrison Avenue (USEPA 2002). Manufacturing activities in Area 11 included the production paint (Rockford Coatings Corporation), furniture varnish (Rockford Varnish Company), and gears and rollers for newspaper presses (Rockwell International Graphics). A restaurant (Villa Di Roma Restaurant), a custom metals finishing company (Accurate Metals Illinois), and a jewelry store are currently located within Area 11 (USEPA 2002).

Light non-aqueous phase liquid (LNAPL) was identified to thicknesses of 25 feet in the 1995 remedial investigation (RI) (Camp, Dresser & McKee [CDM] 1995). Historically chlorinated solvents were stored in eight aboveground storage tanks at Rockford Varnish Company and a dumpster at Rockwell International Graphics, which reportedly leaked cutting fluids into the subsurface. A bunker located in the railroad right-of way reported to be used by the Rockwell Varnish Company has historically seeped a tar-like substance. Rockwell International Graphics also used TCA to clean rollers at its facility until 1983 (USEPA 2002).

10.3 Observations from Site Reconnaissance Reports

Five-year review reports for the site were completed in November 1998, May 2001, May 2003, and May 2013. As part of the 5-year reviews, site visits were conducted to assess site conditions so that USEPA may determine the protectiveness status of the remedies.

10.4 A Synopsis of Secondary Data or Information from Site Reports

The site has a history of industrial activity extending back to approximately 1926. Groundwater investigations in southeast Rockford, performed by the State of Illinois between 1981 and 1988, indicated that many private and municipal wells were impacted by chlorinated solvent contamination at levels exceeding federal health standards. Further investigations determined that solvents were used by industries and were released directly into the environment from storage tanks and improper disposal practices.

The site was added to the National Priorities List on March 1, 1989. USEPA and IEPA have worked throughout the years to connect residents with VOC concentrations exceeding drinking water standards in their wells to the Rockford municipal water supply. USEPA and IEPA are also working on source area treatment within the site.

Between 1991 and 1994, an RI was performed by CDM to define the nature and extent of groundwater contamination. Results of the RI were presented in 1995 in the *Southeast Rockford Final Remedial*

Investigation Report (CDM 1995). In 1996, additional field activities were conducted to investigate the nature and extent of groundwater contamination within source Areas 4, 7, 9/10 and 11 (CDM 2000). Remediation activities completed to date in each of the source areas is presented in the *Fourth Five-Year Review Report* (USEPA 2013) and is summarized in the following subsection. In 2012, S.S. Papadopoulos & Associates, Inc., compiled groundwater analytical results from samples collected between 1988 and 2012 and completed a statistical analysis of chemical concentrations in the groundwater plume (S.S. Papadopoulos 2012). Sitewide groundwater sampling is conducted semiannually by Nationwide Environmental Services, Inc., for 35 groundwater monitoring wells to monitor changes in site groundwater concentrations over time.

10.4.1 Soil and Groundwater Investigations/Remediation in Source Areas

10.4.1.1 Area 4

Elevated concentrations of chlorinated VOCs were detected in soil and groundwater at Area 4 (USEPA 2002). Remediation conducted at Area 4 included the following:

- Contaminated surface soil was removed in September 2005, to prevent direct contact exposures.
- In 2010, a leachate extraction and granulated activated carbon treatment system was installed at the site to treat groundwater contamination.
- IEPA is currently completing a design for an *in situ* thermal soil remedy using electrical resistance heating, to treat remaining contaminated soil at the site. The soil treatment was anticipated to be implemented in 2013 (USEPA 2013).

10.4.1.2 Area 7

Elevated concentrations of ethylbenzene, toluene, and xylenes and chlorinated VOCs were detected in soil in the northern portion of Area 7. Chlorinated VOCs were also detected in shallow groundwater and within the surface water of an unnamed creek (USEPA 2002). Remediation conducted at Area 7 included the following:

- A declaration of restrictive covenants was filed to restrict soil and groundwater use.
- Between 2010 and 2011, 5,372 tons of contaminated soil were excavated for offsite disposal.
- A groundwater extraction and treatment system has been designed to treat contaminated groundwater in the source area. The treatment plant construction is anticipated to be completed in 2014 (USEPA 2013).

10.4.1.3 Area 9/10

Elevated concentrations of VOCs were detected in soil and groundwater at Area 9/10. Remediation conducted at Area 9/10 included the following:

- An air sparge and soil vapor extraction system (AS/SVE) began operation at the site in December 2009. An estimated 1,711 pounds of VOCs were removed through September 2012.
- In November 2010, 734 tons of contaminated soil was excavated and disposed of offsite.
- In June 2012, soil mixing using sodium permanganate and in situ injection of sodium permanganate was performed at the site to reduce in situ contaminant (USEPA 2013).

10.4.1.4 Area 11

Sampling activities conducted from 2007–2008 indicated that elevated concentrations of aromatics (ethylbenzene, toluene, and xylenes) are present in the saturated zone of Area 11; however, they are not found in the vadose zone. Contaminated soil has not been identified at the site. The investigations performed at Area 11 included the following:

- Two membrane interface probe (MIP) investigations have been conducted in Area 11, the first investigation in 2007–2008 and in 2010.

- A two-year groundwater study is currently being conducted by IEPA to evaluate potential site remedies (USEPA 2013).

10.4.2 Historical Vapor Intrusion Investigations

Soil gas surveys were conducted by CDM during Phase I and Phase II site investigations between 1991 and 1993, and additional soil gas sampling was conducted in 1996 within Areas 4, 7, 9/10 and 11. Residential indoor air sampling was conducted during the RI in Areas 4 and 7, and additional indoor air sampling was conducted in 2002 in Areas 4 and 7. The historical soil gas and indoor air data will not be used during the planned soil gas evaluation as soil gas conditions have likely changed over the past 20 years due to contaminant migration and the remedial actions described above. The historical soil gas data may no longer be representative of the current site conditions. However, it may be noted that TCA, trichloroethene (TCE), and PCE were frequently detected in soil gas samples collected in Areas 4, 7, and 9/10, and that mitigation of vapor intrusion was not recommended at any of the buildings sampled based on the indoor air results. The following reports contain the historical vapor intrusion investigation results:

- *Southeast Rockford Final Remedial Investigation* Report (CDM 1995)
- *Final Remedial Investigation Report for the Southeast Rockford Source Control Operable Unit* (CDM 2000)
- *Technical Memorandum: Southeast Rockford Indoor Air Sampling Study* (CDM 2004)

10.5 The Rationale for Inclusion of Chemical and Nonchemical Analyses

The contaminants considered in the design of the soil gas and surface water/pore water sampling program are select VOCs. The select VOCs include: 1,1-dichloroethene, 1,2-dichloroethane, cis-1,2-dichloroethene, trans-1,2-dichloroethene, benzene, ethylbenzene, toluene, xylenes, PCE, TCA, 1,1,2-trichloroethane, TCE, vinyl chloride, and methylene chloride, which are the groundwater contaminants of concern (COCs) identified in the ROD, and 1,1-dichloroethane, which is a daughter product of 1,1,1-trichloroethane and TCE. Soil gas and surface water/pore water will be analyzed for these select VOCs to resolve data gaps identified in the *Fourth Five-Year Review Report for Southeast Rockford Groundwater Contamination Superfund Site* (USEPA 2013). Surface water and pore water samples will also be analyzed for hardness to evaluate the fate and transport of contaminants with regard to ecological receptors.

The investigation will evaluate VOC concentrations in deep soil gas within residential and commercial areas of the site and surface water/pore water within the Rock River. The activities to collect the additional data will consist of the following:

- Review of historical documents to obtain groundwater depth and site lithology, which will be used to select soil gas sampling depths
- Site reconnaissance to evaluate each proposed soil gas sample location. Site reconnaissance activities will include the documentation of physical building characteristics (for example, building proximity, structure type, occupancy status, and air exchange) near each sample location and potential preferential soil gas migration pathways (for example, underground utility conduits)
- Soil gas sample collection and analysis
- Colocated surface water/pore water sample collection and analysis

10.6 Information Concerning Various Environmental Indicators

In conjunction with the *Fourth Five-Year Review Report*, representatives for USEPA, USEPA's contractors, IEPA, IEPA's contractors, and the City of Rockford conducted a site inspection in October 2012.

The purpose of the inspection was to assess conditions so that USEPA may determine the protectiveness status of the remedy for the site. During the inspection a visual evaluation led by the treatment system's operator was conducted for each of the four source areas, a subset of the monitoring well network was inspected, and the current and planned actions for each source area was reviewed. The inspection concluded that all source area facilities and monitoring wells inspected were in good condition. During the inspection, it was discussed that residences that have refused hook-up to the municipal water supply need continued monitoring. IEPA indicated that notifications will periodically be mailed to these residents, and when these properties are sold to ensure the new owner agrees to connect to the municipal water supply.

During preparation of the *Fourth Five-Year Review Report*, interviews were conducted from January to February 2013 with property owners of the source areas, health agencies, and regulatory agencies. The purpose of the interviews was to document any perceived problems or successes with the remedies that have been implemented to-date. The conclusion of the interviews was that no major issues were identified, however a concern was expressed over residents who continue to use private water despite potential health risks. Future actions on the site included the suggestion to periodically sample residential wells, and to update the groundwater model for the site.

For a more thorough description of the environmental site conditions, please refer to the *Fourth Five-Year Review Report for Southeast Rockford Groundwater Contamination Superfund Site* (USEPA 2013).

10.7 Project Decision Conditions ("If..., then..." statements)

Refer to Table 11-1, which summarizes the data quality objectives (DQOs).

Worksheet #11—Project/Data Quality Objectives

TABLE 11-1
Data Quality Objectives

DQO #	Step 1: Statement of Problem	Step 2: Identify Goals of the Study	Step 3: Identify Information Inputs	Step 4: Define Boundary Studies	Step 5: Develop Analytical Approach	Step 6: Specify performance or acceptance criteria	Step 7: Develop plan for obtaining data
1	Previous investigations have identified groundwater contamination underlying residential and commercial properties that could potentially impact humans through the vapor intrusion pathway.	The goal is to determine whether contaminants are present in deep soil gas at concentrations above USEPA screening levels.	Collect deep soil gas samples from selected residential and commercial areas.	The soil gas probe locations will be placed within the extent of the known groundwater plume (more than 10 micrograms per liter [µg/L]) and in areas of suspected residual vadose zone contamination. Soil gas sampling depths will be based on historical groundwater level data. Soil macrocores will also be collected to obtain site lithology and depth to groundwater data to fill data gaps throughout the site.	Analytical parameters were developed based on the fourth 5-year review report and results of previous investigations. Refer to Worksheet #17.	Refer to Worksheets #24, #28, and #36 for acceptance and performance criteria. Laboratory data are considered usable if data validation criteria are met (refer to Worksheet #37 for data usability criteria).	Refer to Worksheet #17 for details on the sample collection design and rationale.
2	Previous investigations have identified contaminated groundwater may be venting to the Rock River and potentially impacting ecological receptors within the river.	The goal is to conduct a phased pore water study using the Trident Probe system: Phase 1 – Sitewide survey of surface water/pore water conditions of the Rock River to identify candidate areas of groundwater venting from the site. Phase 2 – Collect colocated surface water/pore water samples from 9 site and 1 background area for confirmation chemical analyses of groundwater COCs and hardness.	Phase 1 – Sitewide colocated pore water and surface water quality measurements (temperature and conductivity) adjacent to the groundwater plume. Phase 2 – Follow-up and expanded water quality measurements (temperature, conductivity, pH, oxidation-reduction potential [ORP], and total dissolved solids [TDS]) and laboratory analysis of select VOCs for colocated pore water and surface water samples at 10 Phase 1 locations (9 site and 1 background).	The pore water and surface water locations will be positioned within 60, evenly spaced 100-foot long transects extending out from the eastern shore of the Rock River. Transects will be placed within the portion of the river that may be impacted by the groundwater plume (more than 10 µg/L).	The pore water and surface water parameters were selected to identify areas of groundwater venting to surface water. Refer to Worksheet #17.	Performance criteria: Worksheets #24, #28, and #36 and Appendix D (Trident Probe field operating procedures [FOP]) Laboratory data are considered usable if data validation criteria are met (refer to Worksheet #37 for data usability criteria).	Refer to Worksheet #17 for details on the survey design and rationale.

11.1 Who will use the data?

The data will be used by CH2M HILL, USEPA, and IEPA.

11.2 What will the data be used for?

Soil gas, pore water and surface water sample collection and other associated technical assistance tasks will be conducted to fill data gaps necessary to evaluate the nature and extent of contaminants, assess whether further vapor intrusion studies are needed, and assess potential impacts on ecological receptors. The data collected as part of the investigation will be used to meet the DQOs.

11.3 What types of data are needed?

Technical Assistance Task	Sampling Activity/Objective	Sampling Frequency/ Duration	Matrix	Parameters
Soil Gas	Soil gas sampling (Refer to DQO #1)	Once	Soil Gas	Select VOCs
Pore Water	Pore water sampling (Refer to DQO #2)	Once	Pore Water	Select VOCs and hardness
Surface Water	Surface water sampling (Refer to DQO #2)	Once	Surface Water	Select VOCs and hardness

11.4 How "good" do the data need to be in order to support the environmental decision?

The data should meet the project action levels as specified in QAPP Worksheet #15-1 through #15-3, and the QC requirements that are explained in QAPP Worksheet #37.

11.5 How many data are needed? (number of samples for each analytical group, matrix, and concentration)

Worksheet #17 (Sampling Design and Rationale) describes the field investigation activities. Worksheet #18 (Sampling Locations and Methods) summarizes the number of samples and the analytical parameters.

11.6 Where, when, and how should the data be collected/generated?

Detailed information on where, when, and how the data will be collected is provided in Worksheets #17 and 18.

11.7 Who will collect and generate the data?

For soil gas sampling, CH2M HILL will collect environmental samples. For surface water/pore water sampling, the Trident Probe operator, Coastal Monitoring Associates (CMA), will provide environmental samples to CH2M HILL. TestAmerica, Inc., will generate the data for all samples.

11.8 How will the data be reported?

The data will be reported in accordance with procedures outlined in Worksheet #36. Electronic (such as database management system and GIS) data will be stored by CH2M HILL for 7 years after project completion.

11.9 How will the data be archived?

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities. CH2M HILL will be the custodian of the evidence file and will maintain the

contents of the evidence files for the project, including relevant records, reports, logs, field notebooks, sketches, pictures, contractor reports, and data reviews, in a secured area with limited access. CH2M HILL will keep all records for 7 years after project completion. As necessary, records may be transferred to an offsite records storage facility. The records storage facility must provide secure, controlled access records storage.

Worksheet #12—Measurement Performance Criteria

The measurement performance criteria (MPC) for chemical analyses being performed for each matrix and analytical parameter are summarized in Table 12-1 on the following page. The MPCs follow those defined in the referenced USEPA method or laboratory standard operating procedures (SOPs). The quality of the data to be collected for this project will be verified through appropriate MPCs established for both sampling procedures and analytical methods. The criteria relate to data quality indicators (DQIs), consisting of precision, accuracy, representativeness, comparability, completeness, and sensitivity, commonly referred to as PARCCS parameters. The DQIs are defined as follows:

- **Precision** refers to the reproducibility of measurements. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms.
- **Accuracy** refers to the degree of agreement between an observed value (such as sample results) and an accepted reference value. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits.
- **Representativeness** describes the extent to which a sampling design adequately reflects the environmental conditions of a site. Representativeness is determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, operations process locations, and sampling locations.
- **Comparability** addresses the degree to which different methods or data agree or can be represented as similar. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats.
- **Completeness** is a measure of the amount of valid data collected using a measurement system. Completeness is expressed as a percentage of the number of measurements that are specified in this Uniform Federal Policy QAPP.
- **Sensitivity** is the ability of a method or instrument to detect the target analytes at the level of interest. Sensitivity can be measured by calculating the percent recovery of the analytes at the detection limit, which is the minimum concentration of an analyte that can be routinely identified and quantified above the method detection limit by a laboratory.

The quality of the sampling procedures and laboratory results will be evaluated for compliance with project DQOs through a review of overall PARCCs, in accordance with procedures described in Worksheet #37 (Data Usability Assessment). The results will be summarized in an overall data usability report.

Worksheet #12-1—Measurement Performance Criteria Table

Matrix Soil Gas

Analytical Group Select VOCs

Concentration Level Low to High

Sampling Procedure[a]	Analytical Method/SOP[b]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S & A)
FOP #1 and #2	Laboratory SOP #1	Precision	RPD ± 30%	FD	S & A
		Accuracy/Bias	70-130% Recovery	LCS	A
		Completeness	> 90% Laboratory Analysis	Percent Completeness	S & A
		Representativeness	Contamination of sample	MB	S & A
		Comparability	Qualitative measure for field sampling procedures	LCS	A
		Sensitivity	Evidence of shift in instrument response or zero setting MDLs	LCS, IC, CC	A
		Completeness	See Worksheet #37	See Worksheet #37	S & A

[a] Reference number from QAPP Worksheet #21
[b] Reference number from QAPP Worksheet #23
FOP = field operating procedure, SOP = standard operating procedure, VOCs = volatile organic compounds, FD = field duplicate, LCS = laboratory control sample, MB = method blank, FB = field blank, EB = equipment blank, RPD = relative percent difference, MDL = method detection limit, IC = initial calibration, CC = continuing calibration

Worksheet #12-2—Measurement Performance Criteria Table

Matrix Pore Water and Surface Water

Analytical Group Select VOCs

Concentration Level Low

Sampling Procedure[a]	Analytical Method/SOP[b]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S & A)
Appendix D	Laboratory SOP #2	Precision	RPD ± 30%	MS/MSD, FD	S & A
		Accuracy/Bias	± Percent Recovery (Varies based on analyte)	LCS, MS/MSD	A
		Completeness	> 90% Laboratory Analysis	Percent Completeness	S & A
		Representativeness	Contamination of sample	MB, FB, EB	S & A
		Comparability	Qualitative measure for field sampling procedures	LCS, MS/MSD	A
		Sensitivity	Evidence of shift in instrument response or zero setting MDLs	LCS, IC, CC	A
		Completeness	See Worksheet #37	See Worksheet #37	S & A

[a] Reference number from QAPP Worksheet #21
[b] Reference number from QAPP Worksheet #23
FOP = field operating procedure, SOP = standard operating procedure, VOCs = volatile organic compounds, FD = field duplicate, LCS = laboratory control sample, MS = matrix spike, MSD = matrix spike duplicate, MB = method blank, FB = field blank, EB = equipment blank, RPD = relative percent difference, MDL = method detection limit, IC = initial calibration, CC = continuing calibration

Worksheet #12-3—Measurement Performance Criteria Table

Matrix Pore Water and Surface Water

Analytical Group Hardness

Concentration Level Low

Sampling Procedure[a]	Analytical Method/SOP[b]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S & A)
Appendix D	Laboratory SOP #3 and Laboratory SOP #4	Accuracy/Bias	70-130% Recovery	LCS	A
		Completeness	> 90% Laboratory Analysis	Percent Completeness	S & A
		Representativeness	Contamination of sample	MB	S & A
		Comparability	Qualitative measure for field sampling procedures	LCS	A
		Sensitivity	Evidence of shift in instrument response or zero setting MDLs	LCS, IC, CC	A
		Completeness	See Worksheet #37	See Worksheet #37	S & A

[a] Reference number from QAPP Worksheet #21
[b] Reference number from QAPP Worksheet #23
FOP = field operating procedure, SOP = standard operating procedure, LCS = laboratory control sample, MB = method blank, MDL = method detection limit, IC = initial calibration, CC = continuing calibration

Worksheet #13—Secondary Data Uses and Limitations

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Sitewide RI Report	*Southeast Rockford Final RI Report* (CDM 1995)	CDM	Well construction data, groundwater contours, and contaminant concentrations will be used to refine the soil gas and pore water sampling locations.	None
RI Report for Areas 4, 7, 9/10, and 11	*Final Remedial Investigation Report for the Southeast Rockford Source Control Operable Unit* (CDM 2000)	CDM	Well construction data, groundwater contours, and contaminant concentrations will be used to refine the soil gas sampling locations.	None
Five-Year Review Report	*Fourth Five Year Review Report, Southeast Rockford Groundwater Contamination Superfund Site, Winnebago County, Illinois* (USEPA 2013)	USEPA	Historical data summary and recommendations for soil gas and pore water investigations.	None
Compilation of all Southeast Rockford site groundwater monitoring data; estimated groundwater plume map	*Statistical Analysis of Chemicals Concentrations in Groundwater and Mapping* (S.S. Papadopoulos & Associates, Inc. 2012) and associated database	S.S. Papadopoulos & Associates, Inc.	The extent of the groundwater plume presented in this report will be used to select soil gas and pore water sampling locations. Well construction data will be used to select the monitoring wells screened within the shallow groundwater interval as water level data from these wells will be used to determine the depth of the proposed soil gas probes.	Well construction data were not available for all site monitoring wells.
2012 Area 4 groundwater monitoring data	*Revised Source Area 4 Groundwater Management Zone 2012 Annual Report* (CDM Smith 2013)	CDM Smith	2012 water level data will be used to determine the depth of the proposed soil gas probes.	A limited number of shallow monitoring wells (7) were gauged throughout the site in 2012; therefore, the estimated depth to water will need to be interpolated in portions of the site.
2012 Area 9/10 groundwater monitoring data	*2012 Annual GMZ Monitoring and System Performance Report* (Stantec 2013)	Stantec	2012 water level data will be used to determine the depth of the proposed soil gas probes.	
2011 site-wide groundwater monitoring data	*Southeast Rockford Groundwater NPL Site Ground Water Monitoring Report Semi-Annual Monitoring Event--December* (National Environmental Services Inc. 2012)	National Environmental Services, Inc.	2011 water level data will be used to determine the depth of the proposed soil gas probes.	The groundwater surface did not intersect the monitoring well screens at any of the 7 shallow monitoring zones.

Worksheets #14 and #16—Project Tasks and Schedule

14.1 Sampling Tasks

Applicable field operating procedures (FOPs) for the project tasks outlined in this worksheet are listed on Worksheet #21 and in Appendix A. The sampling tasks are described in Worksheets #17 and 18.

14.2 Analysis Tasks

TestAmerica, Inc., will analyze soil gas samples for VOCs and pore water and surface water samples for VOCs and hardness.

The specific methods for these analyses are found in Worksheet #23.

14.3 Quality Control Tasks

Implement FOPs. See Worksheets #11, #12-1, #12-2, #12-3, #15-1, #15-2, #15-3, #22, #24, #25, #27, #28-1, #28-2, #28-3, #28-4, #28-5, #28-6, and #28-7 for items related to QC. QC samples are described in Worksheet #28.

14.4 Secondary Data

See QAPP Worksheet #13.

14.5 Data Management Tasks

The following are the team members and their responsibilities for the data management process:

- **Project Chemist—**Responsible for providing weekly the chain-of-custody forms and establishing the sample tracking system. Oversees proper use of USEPA's sample management system (Scribe) and accuracy of the information entered. Reviews laboratory data for accuracy and quality and compares electronic outputs for accuracy to laboratory electronic copies. Conducts tracking of samples, forwards tracking information and received data to the database manager, and identifies the data inputs (for example, sample numbers) to use in generating tables and plots.
- **Database Manager—**Responsible for setting up the data management system in consultation with the project chemist at the beginning of the data evaluation task. Also oversees the data management process, including data conversion/manual entry into the data management system, QC of the entered data, and preparation of the required tables and plots of the data. Coordinates with the person responsible for reviewing the entered data for QC purposes. Forwards all deliverables to the site manager.
- **GIS Manager—**Responsible for coordinating with the site manager to set up the geodatabase prior to sampling. Maintains spatial layers and overall geodatabase integrity and accuracy. Provides all GIS-related outputs for reports.

Sample Tracking

The project chemist is responsible for tracking samples in the sample tracking database to ensure that the analytical results for all samples sent for analysis are received. Copies of chains of custody from the field team are used to enter in sample IDs, collect date, and analyses. Upon receipt of a sample receipt notice from the laboratory, the date received by the laboratory, and a date the electronic copy is due will be entered. Likewise, upon receipt of the electronic copy and electronic data deliverable (EDD), the date they were received will also be entered. The EDDs will be uploaded when received from the laboratory and will

be tracked in the sample tracking table. Validation qualifiers will be added to the database and the results will be qualified accordingly.

Data Types

The data will be added to the project database as they become available. The data will include new data collected in the laboratory and validated by CH2M HILL. The data source will be noted in the database.

Data Tracking and Management

Every data set received from analytical laboratories will be tracked individually. Analytical laboratory reports of chemical analysis results will be tracked in a consistent fashion. Every data set will be assigned a unique identifier. The date of receipt, status of data validation, and status of database entry for each data set will all be tracked and recorded in the project database.

Hard/Electronic Copy

Measurements made during field data collection activities will be recorded in field logbooks and sample processing logs. Field data will be reduced and summarized, tabulated, and stored along with the field logbooks and sample processing logs.

All raw analytical laboratory data are stored electronically.

Data Input Procedures

Sampling information, analytical results, applicable QA/QC data, data validation qualifiers, and other field-related information will be entered into the project database for storage and retrieval during data evaluation and report development. The analytical data will be loaded into the database using EDD files received from the analytical laboratory. Validation qualifiers will be entered manually. Other available field-related data collected will be manually entered onto standard EDD templates for loading into the database. Historical data, either in hard copy or electronic form, will be manually entered on or formatted to standard EDD templates for database loading.

Computer Database

The technical data, field observations, laboratory analytical results, and analytical data validation will be managed using EQuIS, a third-party database system by Earthsoft, Inc., that is used in USEPA Region 5 to store and analyze project data submissions. The core EQuIS applications are its chemistry and geology modules, each of which is associated with its own underlying Microsoft Access database. CH2M HILL owns licenses for the geology and chemistry modules. The EQuIS database system is based on a relational model in which independent tables, each containing a certain type or entity of data, can be linked through selected fields that are common to two or more tables. The database design allows for the inclusion of historical data, and allows users to effectively conduct trend analysis and generate a variety of data reports to aid in data interpretation.

The database must be protected from unauthorized access, tampering, accidental deletions or additions, and data or program loss that can result from power outages or hardware failure. The following procedures will be adopted to ensure protection:

- The master database will be stored on a network file server local to the installation of the EQuIS data management system. Members of the data management team involved in loading, modifying, or querying the database will be given access through EQuIS user accounts and passwords, as well as the appropriate network server permissions.
- Copies of the master database will be stored on the local area network for access by project staff through reporting tools developed to minimize possible database corruption by users. Whenever the

master database is updated or modified, it will be recopied to the local area network to ensure that the current copy is available to users.

- Daily backups of the master database and its copies will be made to ensure that the data will not be lost due to problems with the network.

GIS Description

A project geodatabase will be agreed upon and set up prior to sampling by the site manager, database manager, and GIS manager. Workflow for creating, maintaining, and organizing geospatial data will follow the Spatial Data Standard format for projects whenever possible.

An ArcView project or extension will be used providing the following functionality: load and display project site base maps; display sampling station locations and associated sampling data (date, media, and results); and perform ad hoc queries to highlight sampling locations meeting user-entered criteria for sampling (for example, data by date, sample type, analyte, depth/elevation, result value, or any combination thereof). Results will be shown as stations highlighted on the map.

Documentation

Documentation of data management activities is critical because it provides the following:

- An electronic copy record of project data management activities
- Reference information critical for database users
- Evidence that the activities have been properly planned, executed, and verified
- Continuity of data management operations when personnel changes occur

The data management plan will serve as the initial general documentation of the project data management efforts. Additional documentation will be maintained to document specific issues such as database structure definitions, database inventories, database maintenance, user requests, database issues and problems, and client contact.

Evidence File

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities. CH2M HILL is the custodian of the evidence file and maintains the contents of the evidence files for the project, including all relevant records, reports, logs, field notebooks, sketches, pictures, contractor reports, and data reviews in a secured area with limited access.

CH2M HILL will keep all records until project completion and closeout. As necessary, records may be transferred to an offsite records storage facility. The records storage facility must provide secure, controlled-access records storage. Records of raw analytical laboratory data, QA data, and reports will be kept by TestAmerica, Inc., for at least 7 years.

Presentation of Study Data

Depending on data user needs, data presentation may consist of any of the following formats:

- Tabulated results of data summaries or raw data
- Figures showing concentration isopleths or location-specific concentrations
- Tables providing statistical evaluation or calculation results
- Presentation tools, such as ARCINFO or similar analysis/presentation aids

In addition to laboratory data, other physical data will be collected during field efforts. The information will be stored in the project database. Other types of data elements may be added as the field investigation needs and activities evolve.

14.6 Assessment and Audit Tasks

See Worksheets #31, #32, and #33.

14.7 Data Review Tasks

The laboratory will make sure the data are complete for all samples received. TestAmerica, Inc., data will be validated by CH2M HILL using the National Functional Guidelines, laboratory SOPs, and the QAPP.

Validated data and field logs will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Final data are placed in the database with qualifiers.

See Worksheets #34 though #37 for the tasks.

14.8 Documentation and Records

Records and field measurements of all samples will be collected in notebooks. Chains of custody, air bills, and sample logs will be prepared and retained for each sample.

A copy of the final QAPP will be kept at the CH2M HILL Milwaukee office.

Project Schedule

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Site-specific Plans	CH2M HILL	November 2013	November 2013	Site-specific plans	November 2013
Sample Collection	CH2M HILL	TBD	TBD	Samples to laboratory	NA
Laboratory Analysis	TestAmerica, Inc.	Within required analytical holding times	21 business days	Data package	21 business days for all other properties
Data Validation	CH2M HILL	After receipt of analytical data reports	21 business days after receipt of final data	Data validation report	21 business days after receipt of final data
Data Evaluation	CH2M HILL	After receipt of final data	45 business days after receipt of validated data	Pore water technical memorandum and soil gas technical memorandum	45 business days after receipt of validated data

NA = not applicable

Worksheet #15-1—Project Action Limits and Laboratory-Specific Detection/Quantitation

Matrix: Soil Gas

Analytical Group: VOCs

Concentration Level: Low - High

Analyte	CAS Number	Project Action Limit[a] (µg/m³)	Achievable Laboratory Limits	
			MDLs (µg/m³)	RLs (µg/m³)
1,1,1-Trichloroethane	71-55-6	52,143	0.11	1.1
1,1,2-Trichloroethane	79-00-5	2.1	0.087	1.1
1,1-Dichloroethane	75-34-3	150	0.095	0.41
1,1-Dichloroethene	75-35-4	2,086	0.34	0.79
1,2-Dichloroethane	107-06-2	9.4	0.073	0.81
1,2-Dichloroethene (mixed isomers)	540-59-0	626	0.091	0.79
Benzene	71-43-2	31	0.058	0.64
Ethylbenzene	100-41-4	97	0.065	0.87
Methylene Chloride	75-09-2	6300	0.08	1.7
Tetrachloroethene	127-18-4	420	0.10	1.4
Toluene	108-88-3	52,143	0.053	0.75
Trichloroethene	79-01-6	21	0.049	1.1
Vinyl Chloride	75-01-4	16	0.023	0.51
Xylenes (Total)	1330-20-7	1,043	0.069	0.87

VOCs = volatile organic compounds, MDL = method detection limit, RL = reporting limit, µg/m³ = microgram per cubic meter
[a] USEPA Vapor Intrusion Screening Level (VISL) Calculator Version 3.1, November 2013 Regional Screening Levels (RSLs) (Hazard Index = 1; Carcinogenic Risk = 1E-05)

Worksheet #15-2—Project Action Limits and Laboratory-Specific Detection/Quantitation

Matrix: Pore Water and Surface Water

Analytical Group: VOCs

Concentration Level: Low

			Achievable Laboratory Limits	
Analyte	**CAS Number**	**Project Action Limit[a] (µg/L)**	**MDLs (µg/L)**	**RLs (µg/L)**
1,1,1-Trichloroethane	71-55-6	11	0.20	1.0
1,1,2-Trichloroethane	79-00-5	1,200	0.28	1.0
1,1-Dichloroethane	75-34-3	47	0.19	1.0
1,1-Dichloroethene	75-35-4	25	0.31	1.0
1,2-Dichloroethane	107-06-2	100	0.28	1.0
1,2-Dichloroethene (mixed isomers)	540-59-0	590	0.12	1.0
Benzene	71-43-2	370	0.074	0.50
Ethylbenzene	100-41-4	90	0.13	0.50
Methylene Chloride	75-09-2	98.1	0.68	5.0
Tetrachloroethene	127-18-4	111	0.17	1.0
Toluene	108-88-3	2	0.11	0.50
Trichloroethene	79-01-6	21	0.19	0.50
Vinyl Chloride	75-01-4	930	0.10	0.50
Xylenes (Total)	1330-20-7	13	0.068	1.0

VOCs = volatile organic compounds, MDL = method detection limit, RL = reporting limit, NA = not applicable, µg/L = microgram per liter

[a] USEPA Ecological Freshwater Screening Levels (http://www.epa.gov/reg3hscd/risk/eco/btag/sbv/fw/screenbench.htm)

Worksheet #15-3—Project Action Limits and Laboratory-Specific Detection/Quantitation

Matrix: Pore Water and Surface

Analytical Group: General

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)	Achievable Laboratory Limits	
			MDLs (mg/L)	RLs (mg/L)
Hardness	NA	NA	0.66	1.32

MDL = method detection limit, RL = reporting limit, NA = not applicable, mg/L = milligrams per liter

Worksheet #17—Sampling Design and Rationale

The *Fourth Five-Year Review Report for the Southeast Rockford Site* (USEPA 2013) identified the need to conduct a deep soil gas investigation to determine whether vapor intrusion has the potential to impact residential and commercial properties above the plume area. Additionally, the 5-year review identified the need to evaluate potential groundwater discharge to the Rock River and recommended a pore water study within the Rock River to evaluate potential impacts to ecological receptors.

The field activities will be conducted according to the FOPs provided in Appendix A. The number of samples and the analytical parameters planned are summarized in Worksheet #18 (Sampling Locations and Methods). A summary of historical site data used to select sampling points is presented in Worksheet # 13.

17.1 Deep Soil Gas Sampling Approach

Fifty permanent soil gas probes will be installed throughout the residential and commercial areas overlying the groundwater plume extent. The permanent soil gas probes will be located within the City of Rockford right-of-way. The City of Rockford will be notified of the planned sampling locations during field planning activities and its concurrence for the locations will be obtained. A review of utility drawings from the City of Rockford will be performed for the proposed soil gas probe locations. The Illinois one-call service will be notified prior to soil boring activities for marking of utilities near each proposed soil gas probe location. A private utility locating subcontractor will also be used to locate underground utilities.

Historical groundwater elevations will be evaluated at each proposed soil gas sampling location. The depth of each soil gas probe will be placed approximately three feet above the highest known groundwater level at that location. To fill data gaps in the groundwater elevation data, 10 soil macrocores will be collected with a direct push rig prior to the soil gas probe installation to evaluate soil lithology and depth to groundwater at proposed sample locations that are not located in close proximity to the existing monitoring wells.

The proposed locations for the 50 permanent soil gas probes and 10 soil cores were placed throughout of the groundwater plume extent, as defined in the *Statistical Analysis of Chemicals Concentrations in Groundwater and Mapping* report (S.S. Papadopoulos 2012) to evaluate deep soil gas VOC concentrations in these areas. A subset of the samples were placed in Areas 4, 7, and 9/10, as elevated contaminant concentrations have been observed in the known source areas. The proposed soil gas probe and soil core locations are presented in Figure 4. The following approach was used when identifying the proposed sampling locations:

- Preference was given to areas where groundwater concentrations exceed USEPA vapor intrusion screening levels (VISLs; MCL-based, if available, or risk-based VISLs using a target risk of 1×10^{-5}) as presented in the fourth 5-year review (USEPA 2013).
- Preference was given to the locations in Area 4 (along Marshall Street) and Area 7 (on Bavarian Lane) where historical soil gas samples exceeded screening levels (as presented in the fourth 5-year review (USEPA 2013).
- Available data from all 37 groundwater monitoring wells (regardless of screened depth), as presented in the fourth 5-year review (USEPA 2013), were used in the VISL comparison.
- Preference was given to known source areas in Areas 4, 7, and 9/10.
- Active remediation systems in Areas 4, 7, and 9/10 were avoided.
- Sampling locations were placed in public right-of-ways.

The permanent soil gas probes will be installed by direct-push technology (DPT) rig at a depth above the capillary fringe (approximately 3 feet above the water table), at an approximate depth of 25 feet below

ground surface (bgs). The actual depth of each probe will be determined based on recent groundwater monitoring data. Additionally, 10 soil borings will be placed throughout the investigation area to a depth of approximately 30 feet bgs to fill data gaps in site lithology and groundwater depth based on a review of available monitoring well data. The soil cores will be colocated with 10 of the 50 permanent soil gas probe locations. Soil cores will be photographed and characterized using the Unified Soil Classification System in accordance with the ASTM International Standard Practice for Description and Identification of Soils (ASTM International D2488) in accordance with FOP 1.

Each permanent soil gas probe will be constructed with an expendable implant anchor, a 21-inch long vapor sampling implant made of stainless-steel wire-mesh screen with a 0.5-inch outer diameter. The screen will be attached to Teflon tubing that will extend to the ground surface. Glass beads will be placed approximately 6 inches above the top of the screen, followed by a mix of powdered bentonite and glass beads to the ground surface. The probes will be completed with 4-inch-diameter locking traffic-rated flush-mount covers secured with cement.

The final location will be documented by global positioning system (GPS). Detailed description of the sample collection method, sample positioning, and core processing and sample preparation is provided in FOPs 1 and 2.

Each soil gas probe will be allowed to equilibrate for at least 24 hours after installation is complete, prior to sampling. Each soil gas probe will be helium leak checked, and three dead volumes of soil gas will be purged prior to sampling. The dead volume will include the probe screen, glass beads, and the probe tubing.

Deep soil gas samples will be collected from each permanent soil gas probes in 1-liter Summa canisters equipped with flow controllers set to 100 milliliters per minute (for a 10-minute sample collection time). The soil gas samples will be analyzed for site-specific VOCs (1,1-dichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene, benzene, ethylbenzene, toluene, xylenes, tetrachloroethene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethene, vinyl chloride, and methylene chloride) by USEPA Method TO-15. The site-specific VOCs were selected based on the VOC constituents identified in the ROD (USEPA 2002).

17.2 Pore Water Investigation

A surface water/pore water investigation will be conducted within the portion of the Rock River that may be impacted by the Southeast Rockford groundwater VOC plume, as defined in the *Statistical Analysis of Chemicals Concentrations in Groundwater and Mapping* report (S.S. Papadopoulos 2012). Using the Trident Probe, the surface water/pore water investigation will monitor for contrasts in temperature and conductivity between surface water and groundwater to determine likely areas of groundwater-to-surface water venting (upwelling, or discharge). At likely venting locations, surface water and pore water samples will be collected and analyzed for VOCs. The following sections describe the approach that will be used for the pore water investigation.

17.2.1 Trident Probe System

Developed by CMA (San Diego, California), the Trident Probe is a direct-push system equipped with specific conductance and temperature (C/T) sensors, and water sampling probes. While the C/T sensors are used to obtain water quality measurements, the sampling probes are used to collect water samples for chemical characterization. The sampling probes are equipped with sand pack pre-filters (well driller's sand), which help with sample development and improve sampling efficiency. Survey/sample locations are recorded with a GPS sensor mounted on the top of the push-pole of the Trident Probe system. For this investigation, the conductivity sensor will be deployed in the "liquid tip" configuration instead of the bulk sediment conductivity sensor. For the liquid tip configuration, the sensor is housed within a secondary screen and responds only to the pore water conductivity, as opposed to bulk sediment interstitial water.

CMA will serve as the subcontractor for the Trident Probe study, providing and running the system during the field investigation. Appendix D provides the CMA SOP and Trident Probe system information.

17.2.2 Approach

The surface water/pore water survey will be conducted with a two-phased approach. Phase 1 will consist of a water quality survey to identify candidate areas of venting. Phase 2 will consist of collecting pore water samples for chemical characterization at locations identified through the Phase 1 results. The following sections describe the procedures that will be employed during each phase.

17.2.2.1 Phase 1 (Survey)

Phase 1 will consist of a transect-based water quality survey whereby conductivity and temperature will be measured and contrasted between colocated surface water and pore water (groundwater) using the Trident Probe system C/T sensors. Sixty survey transects are proposed for Phase 1 (Figure 3). Each transect will run perpendicular to the shore, and measurements will be collected in surface water and pore water at up to 4 discrete points along a transect (25, 50, 75, and 100 feet from shore), for a total of 240 survey points. It is anticipated that pore water will be accessed at 12 inches below sediment surface[1]. The co-located surface water sample will be collected in the water column as close to the sediment surface as possible (estimated within 4 inches) above the location of pore water sample collection. Each measurement/sample point will be marked by GPS coordinates.

At each location, the following procedures will be followed to conduct the Phase 1 survey:

1. Surface water depth (depth of the water column to sediment surface) will be measured and recorded.
2. Trident Probe system will be lowered to the sediment surface such that C/T sensors are within 4 inches of the sediment surface.
3. Surface water C/T will be logged via the Trident Probe data acquisition system and recorded in the field log book.
4. Trident Probe system will be advanced into the sediment to situate probes at 12 inches below sediment surface.
5. Pore water C/T will be logged via the Trident Probe data acquisition system and recorded in the field log book.
6. GPS coordinates will be logged via the Trident Probe data acquisition system and recorded in the field log book.
7. The Trident Probe system will be withdrawn and the boat will be positioned at the next location for Step 1.

Following collection of C/T measurements at all locations, results will be mapped and evaluated to identify likely areas of groundwater venting. Based upon these survey results and maps, CH2M HILL will consult with USEPA to select 10 locations (1 background and 9 plume sample locations), for collection of co-located pore water and surface water samples for chemical analysis during Phase 2.

17.2.2.2 Phase 2 (Sampling)

Phase 2 will be conducted during the same field event as Phase 1. After receiving USEPA concurrence on the 10 colocated surface water/pore water sample locations identified through Phase 1, samples will be collected using the Trident Probe. The samples will be used to evaluate the potential venting of groundwater-based VOCs into the surface water environment (or lack thereof) relative to the pore water

[1] The top 12 inches of sediment is commonly assumed to be the most biologically active zone and also the most likely area of active groundwater-to-surface water communication. The exact sample interval might differ slightly during the field investigation depending on field conditions, but will be deep enough to minimize potential surface water drawdown into the sample interval.

results. Samples will be pulled to the sampling deck of the boat via low-flow peristaltic pump-driven procedures (Appendix D).

During Phase 2, the Phase 1 sample depths for surface water (within 4 inches above sediment surface) and pore water (12 inches below sediment surface) will be used. In addition to in situ surface water and pore water C/T measurements with the Trident Probe sensors, additional water quality measurements will be obtained ex situ after samples are pulled to the boat. Ex situ measurements of conductivity, temperature, pH, oxidation-reduction potential (ORP), and total dissolved solids (TDS) will also be collected using a Myron L Ultrameter. Following water quality measurements, additional volume of surface and pore water will be bottled for submission to an offsite laboratory for chemical analyses.

At each location, the following procedures will be followed to conduct the Phase 2 sampling:

1. Phase 1 coordinates will be used to locate sample location.
2. Surface water depth (depth of the water column to sediment surface) will be measured and recorded.
3. Trident Probe system will be lowered to the sediment surface to situate probes within 4 inches of the sediment surface.
4. Surface water quality measures (both in situ C/T [Trident Probe] and ex situ [Ultrameter parameters]) will be logged or recorded.
5. Surface water samples will be placed into bottle ware for chemical analyses.
6. Trident Probe system will be advanced into the sediment to situate probes at 12 inches below sediment surface.
7. Pore water quality measures (both in situ C/T [Trident Probe] and ex situ [Ultrameter parameters]) will be logged or recorded.
8. Pore water samples will be placed into bottle ware for chemical analyses.
9. GPS coordinates will be logged via the Trident Probe data acquisition system and recorded in the field log book.
10. The Trident Probe system will be withdrawn and boat will be positioned at the next location for Step 1.

All samples will be analyzed for site-specific VOCs (1,1-dichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene, benzene, ethylbenzene, toluene, xylenes, tetrachloroethene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethene, vinyl chloride, and dichloromethane) by USEPA Method SW-846 8260B. The site-specific VOCs were selected based on the VOC constituents identified in the ROD (USEPA 2002). The pore water and surface water samples will also be analyzed for hardness to evaluate fate and transport of the site contaminants.

17.3 Equipment Decontamination

All non-disposable equipment used during the soil gas investigation will be decontaminated by washing with Alconox followed by a water rinse prior to reuse. No decontamination water is anticipated to be generated during the pore water investigation.

17.4 Investigation-derived Waste Characterization and Disposal

Investigation-derived waste (IDW) generated during collection of the soil cores and installation of the permanent soil gas probes will include soil and decontamination water. An estimated 14 drums of waste will be generated during the field investigation activities. IDW will be characterized and disposed of in accordance with local, state, and federal regulations as specified in the site management plan. All IDW will

be held at a staging area located on the Rockford Site. Soil samples will be collected and analyzed for the following: toxicity characteristic leaching procedure (TCLP) VOCs, TCLP semivolatile organic compounds (SVOCs), TCLP Pesticides, TCLP Herbicides, polychlorinated biphenyls (PCBs), TCLP Metals, pH, and flashpoint. Aqueous samples will be collected and analyzed for the following: VOCs, SVOCs, pesticides, herbicides, PCBs, Resource Conservation and Recovery Act (RCRA) metals, pH, and flashpoint.

Worksheet #18—Sampling Locations and Methods

Note: The sample locations and rationale for the proposed sampling event will be finalized following the preliminary field survey. All samples will be analyzed for select VOCs (1,1-dichloroethane, 1,1-dichloroethene, 1,2-dichloroethane, 1,2-dichloroethene, benzene, ethylbenzene, toluene, xylenes, PCE, TCA, 1,1,2-trichloroethane, TCA, vinyl chloride, and methylene chloride). The pore water and surface water samples will also be analyzed for hardness. This worksheet will be completed once locations are identified.

Sampling Location/ID Number	Matrix	Depth (feet below ground surface)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling FOP Reference[a]	Rationale for Sampling Location
Soil Gas Investigation (Figure 4)							
Residential/commercial properties within the site. Unique IDs will include the location.	Soil Gas	Depth is dependent upon the groundwater depth. Each soil gas probe will be set approximately 3 feet above the highest known groundwater level at each probe location. It is estimated that the maximum probe depth will be approximately 40 feet bgs.	Select VOCs	Low - High	50 (5)	FOP #1 and #2	To determine nature and extent of contaminant and the need for further vapor intrusion studies.
Pore Water Investigation (Figure 3)							
Areas of groundwater-to-surface water venting within the Rock River. Unique IDs will include the location.	Pore Water and Surface Water	Pore water will be collected within top 4 inches of the sediment surface. A specific sample interval will be determined during field preparation. The colocated surface water sample will be collected in the water column as close to the sediment surface as possible above the location of pore water sample collection.	Select VOCs and Hardness	Low	10 (1)	Appendix D	To determine nature and extent of contaminant and support the ecological risk assessments.
Investigation-derived Waste Sampling							
IDW-1	Soil	NA	TCLP VOCs, TCLP SVOCs, TCLP pesticides, TCLP herbicides, PCBs, TCLP metals, pH, and flashpoint	Low	1	NA	Waste characterization

Sampling Location/ID Number	Matrix	Depth (feet below ground surface)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling FOP Reference[a]	Rationale for Sampling Location
IDW-2	Aqueous	NA	VOCs, SVOCs, pesticides, herbicides, PCBs, RCRA metals, pH, and flashpoint	Low	1	NA	Waste characterization

[a] Specify the appropriate letter or number for the FOP table (Worksheet #21).

Worksheets #19 and #30—Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Containers (number, size, and type)	Preservation Requirements	Maximum Holding Time (preparation/analysis)	Data Package Turnaround
Environmental Samples							
Soil Gas	Select VOCs	Low - High	Laboratory SOP #1	1-liter Summa Canister	None	30 days	Full Package – 21 business days
Pore Water and Surface Water	Select VOCs	Low	Laboratory SOP #2	Three times 40-milliliter (mL) glass vials	HCl to pH <2; 4 degrees Celsius	14 days to analysis	Full Package – 21 business days
Pore Water and Surface Water	Hardness	Low	Laboratory SOP #3 Laboratory SOP #4	250-mL plastic	HNO_3 to pH < 2, 4 degrees Celsius	180 days to digest and analyze	Full Package – 21 business days
Waste Characterization Samples							
IDW-Soil	TCLP VOCs	Low	Laboratory SOP #12 Laboratory SOP #2	4-ounce or larger wide-mouth glass jar, no head space	4 degrees Celsius	14 days to TCLP, 14 days to analysis	Full Package – 21 business days
IDW-Soil	TCLP SVOCs, TCLP Pesticides, TCLP Herbicides and PCBs	Low	Laboratory SOP #13 Laboratory SOP #14 Laboratory SOP #5 Laboratory SOP #6 Laboratory SOP #7 Laboratory SOP #8	16-ounce or larger wide-mouth glass jar	4 degrees Celsius	14 days to TCLP, 7 days to extract, 40 days to analysis	Full Package – 21 business days
IDW-Soil	TCLP Metals	Low	Laboratory SOP #3 Laboratory SOP #4 Laboratory SOP #9	4-ounce or larger wide-mouth glass jar	4 degrees Celsius	14 days to TCLP, 180 days to digest and analyze (28 days for mercury)	Full Package – 21 business days
IDW-Soil	pH Flashpoint	Low	Laboratory SOP #10 Laboratory SOP #11	4-ounce or larger wide-mouth glass jar	4 degrees Celsius	As soon as possible, 28 days	Full Package – 21 business days
IDW-Aqueous	VOCs	Low	Laboratory SOP #2	Three times 40-mL glass vials	HCl to pH <2; 4 degrees Celsius	14 days to analysis	Full Package – 21 business days
IDW-Aqueous	SVOCs	Low	Laboratory SOP #5	Two times 250-mL amber glass	4 degrees Celsius	7 days to extraction/ 40 days to analysis	Full Package – 21 business days
IDW-Aqueous	Pesticides	Low	Laboratory SOP #6	Two times 125-mL amber glass	4 degrees Celsius	7 days to extraction/ 40 days to analysis	Full Package – 21 business days

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Containers (number, size, and type)	Preservation Requirements	Maximum Holding Time (preparation/analysis)	Data Package Turnaround
IDW-Aqueous	Herbicides	Low	Laboratory SOP #7	Two times 1-liter amber glass	4 degrees Celsius	7 days to extraction/ 40 days to analysis	Full Package – 21 business days
IDW-Aqueous	PCBs	Low	Laboratory SOP #8	Two times 125-mL amber glass	4 degrees Celsius	7 days to extraction/ 40 days to analysis	Full Package – 21 business days
IDW-Aqueous	RCRA Metals (including Mercury)	Low	Laboratory SOP #3 Laboratory SOP #4 Laboratory SOP #9	250-mL plastic	HNO_3 to pH < 2, 4 degrees Celsius	180 days (mercury, 28 days)	Full Package – 21 business days
IDW-Aqueous	pH Flashpoint	Low	Laboratory SOP #10 Laboratory SOP #11	250-mL plastic	4 degrees Celsius	As soon as possible, 28 days	Full Package – 21 business days

[a] Specify the appropriate reference letter or number from the analytical SOPs table (Worksheet #23).

Worksheet #20—Field Quality Control Summary

20.1 Field Duplicate Samples

Field duplicates are two field samples taken concurrently at the same location. They are intended to represent the same population and are taken through all steps of the sampling and analytical procedures in the same manner as the associated native sample. The samples are used to assess the precision of the entire data collection activity, including sampling, sample handling and storage, and site heterogeneity. The field duplicates are assigned a unique sample name and are collected in a separate container from the associated native sample. One field duplicate will be collected for every 10 field samples. During collection of the field duplicate samples of soil gas, two Summa canisters will be connected with a T-connector provided by the laboratory to allow for collection of the parent and duplicate sample at the same time.

20.2 Matrix Spike/Matrix Spike Duplicate Samples

MS/MSD samples are an aliquot of the sample spiked with known concentrations of specific analytes. The spiking occurs before sample preparation and analysis at the laboratory. Samples will be collected in triplicate, and the additional volume used for MS/MSD analysis. An MS/MSD pair will be collected only for the surface water/pore water samples on a frequency of one MS/MSD for every 20 field samples. MS/MSD samples will not be collected for the soil gas sampling effort.

20.3 Field Blanks

During the surface water/pore water sampling effort, field blanks will be collected to assess the potential introduction of contaminants from the surroundings by pouring deionized water directly into the sampling containers while out in normal field conditions. Field blanks will be collected at a frequency of one per week. The field blanks will be analyzed for the same parameters specified for the corresponding matrix. Field blanks will not be collected for the soil gas sampling effort.

20.4 Equipment Blanks

During the surface water/pore water sampling effort, equipment blanks will be collected to assess the potential introduction of contaminants from equipment by pouring deionized water directly onto sampling equipment and then into the sampling containers. Equipment blanks will be collected at a frequency of one per week. The equipment blanks will be analyzed for the same parameters specified for the corresponding matrix. Equipment blanks will not be collected for the soil gas sampling effort.

20.5 Trip Blanks

During the surface water/pore water sampling effort, trip blanks will be used to assess the potential introduction of contaminants to VOC sample containers during field events and during shipment of empty bottles to the site and samples to the laboratory. The trip blank consists of a VOC sample vial filled at the laboratory with laboratory grade deionized water. It is transported to the site in the same manner as the other sampling containers, stored with the samples in the field, and then returned to the laboratory for analysis. One trip blank will be included in each sample cooler containing VOC samples.

Worksheet #20-1—Field Quality Control Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[a]	Number of Samples	Number of FDs	Number of MS/MSDs	Number of FBs/EBs/TBs	Total Number of Samples
Soil Gas	Select VOCs	Low	Laboratory SOP #1	50	5	0	0	55
Pore Water	Select VOCs	Low	Laboratory SOP #2	10	1	1	3	15
Pore Water	Hardness	Low	Laboratory SOP #3 Laboratory SOP #4	10	0	0	0	10
Surface Water	Select VOCs	Low	Laboratory SOP #2	10	1	1	3	15
Surface Water	Hardness	Low	Laboratory SOP #3 Laboratory SOP #4	10	0	0	0	10
IDW-Soil	TCLP VOCs, TCLP SVOCs, TCLP pesticides, TCLP herbicides, PCBs, TCLP metals, pH, and flashpoint	Low	Laboratory SOP #2 Laboratory SOP #3 Laboratory SOP #4 Laboratory SOP #5 Laboratory SOP #6 Laboratory SOP #7 Laboratory SOP #8 Laboratory SOP #9 Laboratory SOP #10 Laboratory SOP #11 Laboratory SOP #12 Laboratory SOP #13 Laboratory SOP #14	1	0	0	1	2

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[a]	Number of Samples	Number of FDs	Number of MS/MSDs	Number of FBs/EBs/TBs	Total Number of Samples
IDW- Aqueous	VOCs, SVOCs, Pesticides, Herbicides, PCBs, RCRA metals, pH, and flashpoint	Low	Laboratory SOP #2 Laboratory SOP #3 Laboratory SOP #4 Laboratory SOP #5 Laboratory SOP #6 Laboratory SOP #7 Laboratory SOP #8 Laboratory SOP #9 Laboratory SOP #10 Laboratory SOP #11	1	0	0	1	2

[a] Specify the appropriate reference letter or number from the analytical SOPs table (Worksheet #23).

Worksheet #21—Field Operating Procedures

Reference Number	Title, Revision, Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
FOP #1	Installation of Permanent Soil Gas Sampling Probes	CH2M HILL	Geoprobe Soil Gas Implant Method or AMS Dedicated Gas Vapor Probe Method	☐	
FOP #2	Collection of Soil Gas Samples from Temporary and Permanent Probes Using Canisters	CH2M HILL	Canister, flow controller and vacuum gauge	☐	
FOP #3	Surface Water Sampling	CH2M HILL	Dip sampler	☐	
FOP #4	Note-taking and Field Logbook	CH2M HILL	Procedural guidance	☐	
FOP #5	Utility Clearance for Intrusive Operations	CH2M HILL	Procedural guidance	☐	
FOP #6	Packing and Shipping of Environmental Samples	CH2M HILL	Procedural guidance	☐	
FOP #7	Sample Handling and Chain-of-Custody Procedures	CH2M HILL	Procedural guidance	☐	
FOP #8	Equipment Decontamination Procedures	CH2M HILL	Procedural guidance	☐	

Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[a]
GPS unit	Initial calibration	Model-specific, per manufacturer's recommendation	Measures *x* and *y* coordinates	Daily	Model-specific, per manufacturer's recommendation	If GPS fails to calibrate, do not use this meter	Field team leader	NA
LandTec GEM2000 Landfill Gas Meter	None	Model-specific, per manufacturer’s recommendation	Detects oxygen, carbon dioxide, and methane	NA	NA	Not applicable	Field team leader	NA
Photoionization Detector (PID)	Initial calibration	Model-specific, per manufacturer's recommendation	Detects gas and vapor analytes	Daily	Model-specific, per manufacturer's recommendation	If PID fails to calibrate, do not use this meter	Field team leader	NA

[a] Refer to the project FOPs table (Worksheet #21).

Worksheet #23—Analytical SOPs

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
Laboratory SOP #1	Detemination of VOCs in Ambient Air by TO-15, Revision 8, 08/16/2012	Definitive	Select VOCs (Soil Gas)	GC/MS	TestAmerica, Inc.	☐
Laboratory SOP #2	Gas Chromatography/Mass Spectrometry-Volatiles by SW-846 8260B, Revision 23, 03/18/2013	Definitive	VOCs (Pore Water, Surface Water, IDW)	GC/MS	TestAmerica, Inc.	☐
Laboratory SOP #3	Metals Digestion by SW-846 3000, Revision 22, 12/19/2012	Definitive	Hardness and Metals (Pore Water, Surface Water, IDW)	ICP-MS	TestAmerica, Inc.	☐
Laboratory SOP #4	Trace Inductively Coupled Argon Plasma by SW-846 6010B (Section 9.4 Calculation for Hardness by SM2340B), Revision 16, 10/3/2013	Definitive	Hardness and Metals (Pore Water, Surface Water, IDW)	ICAP	TestAmerica, Inc.	☐
Laboratory SOP #5	Gas Chromatography/Mass Spectormetry-Semi-Volatiles Analysis of Semi-Volatiles by SW-846 8270D, Revision 4, 11/30/2012	Definitive	SVOCs (IDW)	GC/MS	TestAmerica, Inc.	☐
Laboratory SOP #6	Gas Chromatography-Pesticides by SW-846 8081A and 8081B, Revision 16, 09/27/2013	Definitive	Pesticides (IDW)	GC/ECD	TestAmerica, Inc.	☐
Laboratory SOP #7	Gas Chromatography-Herbicides by SW-846 8151A, Revision 17, 07/31/2013	Definitive	Herbicides (IDW)	GC/ECD	TestAmerica, Inc.	☐
Laboratory SOP #8	Gas Chromatography-PCBs by SW-846 8082 and 8082A, Revision 16, 09/27/2013	Definitive	PCBs (IDW)	GC/ECD	TestAmerica, Inc.	☐
Laboratory SOP #9	Mercury Analysis by SW-846 7470A/7471A/7471B, Revision 18, 08/23/2013	Definitive	Mercury (IDW)	CVAA	TestAmerica, Inc.	☐
Laboratory SOP #10	pH by SW-846 9040B, 9040C, 9045C, 9045D or 9041A, Revision 16, 08/30/2013	Definitive	pH (IDW)	pH meter	TestAmerica, Inc.	☐
Laboratory SOP #11	Flashpoint by SW-846 1010 or 1010A, Revision 16, 11/14/2012	Definitive	Flashpoint (IDW)	Thermometer	TestAmerica, Inc.	☐
Laboratory SOP #12	Toxicity Characteristic Leaching Procedure, Revision 18, 02/06/2013	Definitive	TCLP (IDW)	NA	TestAmerica, Inc.	☐

Reference Number	Title, Revision, Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
Laboratory SOP #13	Semivolatile and Nonvolatile Organic Compounds from a Wastewater or Leachate Matrix using Separatory Funnel Extraction by SW-846 3510C, Revision 13, 02/25/2013	Definitive	TCLP (IDW)	NA	TestAmerica, Inc.	☐
Laboratory SOP #14	Herbicides from a Wastewater or TCLP Extracts by SW-846 8151A	Definitive	TCLP (IDW)	NA	TestAmerica, Inc.	☐

GC/MS = gas chromatograph/mass spectrometry, GC/ECD = gas chromatograph/electron capture detector, ICP-MS =inductively coupled plasma-mass spectrometry, ICAP = inductively coupled argon plasma, CVAA = cold vapor atomic adsorption

Worksheet #24—Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[a]
GC/MS (for VOCs in Soil Gas)	Initial Calibration (IC)	Instrument receipt, instrument change (new column, source cleaning, etc.), when continuing calibration verification (CCV) is out of criteria.	Five-point curve - The percent relative standard deviation (%RSD) for all analytes must be ≤ 30%, with at most two exceptions up to a limit of 40%.	Repeat calibration if criterion is not met.	Analyst	Laboratory SOP #1
	Initial Calibration Verification (ICV)	Once after each IC.	All analytes within 70 to 130 %R.	Correct problem, rerun ICV. If that fails, repeat IC.		
	CCV	Daily.	All project analytes within ± 30% of true value.	Correct problem, rerun CCV. If that fails, repeat IC.		
	BFB Tune	Every 24 hours.	Refer to method for specific ion criteria.	Retune and/or clean source.		
GC/MS (for VOCs)	IC	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	Five-point curve - The average RFs for system performance check compounds (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorobenzene and ≥ 0.10 for chloromethane, 1,1-dichloroethane and bromoform. The %RSD for RFs for calibration check compounds (CCCs) must be ≤ 30%, and one option below must be met: Option 1) %RSD ≤ 15% for all other compounds. If not met: Option 2) Linear least squares regression: correlation coefficient (r) ≥ 0.995. Option 3) Non-linear regression: coefficient of determination (r^2) ≥ 0.99 (6 points for second order).	Repeat calibration if criterion is not met.	Analyst	Laboratory SOP #2
	ICV	Once after each IC.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat IC.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[a]
	CCV	At the beginning of each 12-hour shift immediately after BFB tune.	1) Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform and 1,1-dichloroethane. 2) %Difference/Drift for all target compounds and surrogates: VOCs ≤ 20%D (Note, D = Difference when using RFs, D = Drift when using least squares regression or non-linear calibration.)	Repeat IC and reanalyze all samples analyzed since the last successful calibration verification.		
	BFB Tune	Every 12 hours.	Refer to method for specific ion criteria.	Retune and/or clean source.		
	Retention time window position establishment	Once after each IC for each analyte and surrogate.	Position shall be set using the midpoint standard of the IC curve when IC is performed. On days when IC is not performed, the initial CCV is used.	N/A		
	Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun IC. Flagging criteria are not appropriate.		
GC/MS (for SVOCs)	ICAL	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	Five-point curve - The average RF for SPCCs must be ≥ 0.050; The %RSD for RFs for CCCs must be <30%, and one option below must be met: Option (1) %RSD ≤ 15% for all other compounds. If not met: Option (2) Linear least squares regression: r ≥ 0.995. Option (3) Non-linear regression: $r^2 \geq 0.99$ (6 points for second order).	Repeat calibration if criterion is not met.	Analyst	Laboratory SOP #5
	ICV	Once after each IC.	All analytes within ± 20% of the expected value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat IC.		
	CCV	At the beginning of each 12-hour shift immediately after DFTPP tune.	1) Average RF for SPCCs: SVOCs ≥ 0.050 2) %Difference/Drift for all target compounds and surrogates: VOCs ≤ 20%D (Note, D = Difference when using RFs, D = Drift when using least squares regression or non-linear calibration.)	Repeat IC and reanalyze all samples analyzed since the last successful calibration verification.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[a]
	DFTPP Tune	Every 12 hours.	Must meet the ion abundance criteria required by the method.	Retune and/or clean source.		
	Retention time window position establishment	Once after each IC for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when IC is performed. On days when IC is not performed, the initial CCV is used.	N/A		
	Evaluation of RRT	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun IC. Flagging criteria are not appropriate.		
GC/ECD (for Pesticides)	ICAL	Instrument receipt, major instrument change, when CCV does not meet criteria.	Five-point curve - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: $r^2 \geq 0.99$ (6 points shall be used for second order) Midpoint calibration of Toxaphene; if detected in sample, 6-point calibration is performed.	Repeat IC and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst	Laboratory SOP #6
	ICV	Immediately following IC.	All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat IC.		
	CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D ≤ 20 for both the quantitation and confirmation columns.	Correct problem, then rerun calibration verification. If that fails, repeat IC. Reanalyze samples since last successful calibration verification.		
	Breakdown Check	At beginning of each 12 hour period.	Degradation <15% for both DDT and Endrin.	Correct problem, then repeat breakdown check.		
GC/ECD (for Herbicides)	ICAL	Instrument receipt, major instrument change, when CCV does not meet criteria.	Six-point curve - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995	Repeat IC and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst	Laboratory SOP #7
	ICV	Immediately following IC.	%D ≤ 15	Correct problem, rerun ICV. If that fails, repeat IC.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[a]
	CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D ≤ 15	Correct problem, rerun CCV. If that fails, repeat IC.		
	Retention time window position establishment	With each sample.	RRT of each target analyte within ± 0.66 RRT units.	Correct problem, then rerun IC. Flagging criteria are not appropriate.		
GC/ECD (for PCBs	IC	Instrument receipt, major instrument change, when CCV does not meet criteria.	Six-point curve - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995	Repeat IC and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst	Laboratory SOP #8
	ICV	Immediately following IC.	%D ≤ 20	Correct problem, rerun ICV. If that fails, repeat IC.		
	CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D ≤ 20	Correct problem, rerun CCV. If that fails, repeat IC.		
	Retention time window position establishment	Once after each IC for each analyte and surrogate.	Position shall be set using the midpoint standard of the IC curve when IC is performed. On days when IC is not performed, the initial CCV is used.	N/A		
ICAP (for hardness and metals)	IC	Instrument receipt, major instrument change, when CCV does not meet criteria.	Minimum one high standard and a calibration blank for all analytes. If more than one calibration standard is used, correlation coefficient (r) ≥ 0.995 (r^2>0.990).	Correct problem, then repeat IC. Flagging criteria are not appropriate.		Laboratory SOP #4
	ICV	Once after each IC, prior to beginning a sample run.	All analytes within 90 to 110 %R.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat IC. Flagging criteria are not appropriate.		
	CCV	After every 10 field samples and at the end of the analysis sequence.	All analytes within 90 to 110 %R.	Correct problem, rerun CCV. If that fails, then repeat IC. Reanalyze all samples since the last successful CCV.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[a]
	Linear dynamic range or high-level check standard	Every 6 months	Within ±10% of true value.	N/A		
	Low-level calibration check standard	Daily, after 1-point IC.	Within ±20% of true value.	Correct problem, then reanalyze. Flagging criteria are not appropriate.		
	Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > MDL.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.		
	Interference check solutions (ICSs)	After beginning of the analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < MDL ICS-AB: Within 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.		
CVAA (for mercury)	IC	Instrument receipt, major instrument change, at the start of each day	Five-point curve. If more than one calibration standard is used, correlation coefficient (r) ≥ 0.995 (r^2>0.990).	Correct problem, then repeat IC. Flagging criteria are not appropriate.	Analyst	Laboratory SOP #9
	ICV	Once after each IC, prior to beginning a sample run.	All analytes within 80 to 120 %R.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat IC. Flagging criteria are not appropriate.		
	CCV	After every 10 field samples and at the end of the analysis sequence.	All analytes within 80-120 %R.	Correct problem, rerun CCV. If that fails, then repeat IC. Reanalyze all samples since the last successful CCV.		
	Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > MDL.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.		

[a] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
GC/ECD = gas chromatograph/electron capture detector, GC/MS = gas chromatograph/mass spectrometry, ICAP = inductively coupled argon plasma, CVAA = cold vapor atomic adsorption, SPCC = system performance check compound, IC = initial calibration, ICV = initial calibration verification, CCV = continuing calibration verification, DFTPP = decafluorotriphenylphosphine, BFB = 4-bromoflurobenzene, %D = percent drift, RRT = relative retention time, %RSD = percent relative standard deviation, RF = response factor, CCC = calibration check compounds, ICS = interference check solution

Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection

☑ Worksheet Not Applicable (State Reason)

TestAmerica, Inc., will keep all maintenance, testing, and inspection records on file at the laboratory. The instrument and equipment maintenance, testing, and inspection activities are documented per the SOPs listed in Worksheet #23.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[a]

[a] Specify the appropriate reference letter or number from the analytical SOPs table (Worksheet #23).

Worksheets #26 and #27—Sample Handling, Custody, and Disposal

Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): Field team leader/CH2M HILL
Sample Packaging (Personnel/Organization): Field team leader/CH2M HILL
Coordination of Shipment (Personnel/Organization): Field team leader/CH2M HILL
Type of Shipment/Carrier: Federal Express Overnight
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): TestAmerica, Inc.
Sample Custody and Storage (Personnel/Organization): TestAmerica, Inc.
Sample Preparation (Personnel/Organization): TestAmerica, Inc.
Sample Determinative Analysis (Personnel/Organization): TestAmerica, Inc.
Sample Archiving
Field Sample Storage (No. of days from sample collection): See QAPP Worksheet #19 for allowable holding time
Sample Extract/Digestate Storage (No. of days from extraction/digestion): See QAPP Worksheet #19 for allowable holding time
Sample Disposal
Personnel/Organization: TestAmerica, Inc.
Number of Days from Analysis: The laboratory will retain samples for at least 90 days and sample extracts for at least 30 days, after submittal, pending the need for reanalysis.

26.1 Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory)

Sample handling and chain-of-custody procedures will be performed in accordance with FOP 7, and packaging and shipping of environmental samples will be performed in accordance with FOP 6.

For the surface water/pore water sampling, sample coolers will be shipped to arrive at TestAmerica, Inc., the morning after sampling (priority overnight) or will be sent by a courier to arrive the same day. The laboratory will be notified of the sample shipment and the estimated date of arrival of the samples being delivered.

For the soil gas sampling, soil gas samples will be shipped in boxes provided by the laboratory and will be shipped for 3-day delivery.

Regulations for packaging, marking/labeling, and shipping of hazardous materials and wastes are promulgated by the U.S. Department of Transportation. Air carriers that transport hazardous materials, in particular Federal Express, require compliance with the current edition of the International Air Transport Association Dangerous Goods Regulations, which applies to shipment and transportation of hazardous materials by air carrier. Following current International Air Transport Association regulations will ensure compliance with U.S. Department of Transportation regulations.

26.2 Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal)

Upon sample receipt, the laboratory sample custodian will verify package seals, open the packages, check temperature blanks, record temperatures, verify sample integrity, and inspect contents against chain-of-custody forms. The project chemist will be contacted to seek resolution of any discrepancies between sample containers and chain-of-custody forms through contract-defined channels of communication. Once the shipment and chain-of-custody form are in agreement, the sample custodian will initiate an internal chain-of-custody form, as well as supply the laboratory task manager with a sample acknowledgement letter. When applicable, sample preservation will be checked and pH documented. If the sample temperatures are outside the required range, the laboratory will contact the project chemist as to the proper course of action. Soil gas sample canister vacuums will be checked upon receipt and the vacuum readings will be provided to the project chemist.

Samples will be logged in and assigned a unique laboratory number for each sample, and the number will be used by all laboratory personnel handling samples to ensure all sample information is captured. Analyses required will be specified by codes assigned to samples at login. Labels containing the laboratory sample number are generated and placed on sample bottles.

After the laboratory labels the samples, they will be moved to refrigerators where they will be maintained at 4 degrees Celsius.

When the analyst is ready to prepare and/or analyze the sample(s), an appropriate member of the sample management department will locate the sample(s) in the locked refrigerator, sign and date the internal sample tracking form, and provide the sample(s) to the analyst. When the analyst is finished with the sample(s), unused portions will be returned to an appropriate member of the sample management department for replacement in a secure refrigerator. The analyst will sign and date internal chain-of-custody forms. In the event that entire samples are depleted during analysis, a notation of “sample depleted” or “entire sample used” will be written on the internal chain-of-custody forms.

Samples will be stored in designated secure, refrigerated storage areas. Samples and sample extracts will be maintained in a secure storage until disposal. No samples or extracts will be disposed of without prior written approval from an appropriate member of the project team. The sample custodian will note the sample disposal date in the sample ledger. The laboratory will dispose of samples in accordance with applicable regulations.

Documentation will be placed in a single, secured project file maintained by the laboratory project manager. The file will consist of the following components: agreements, correspondence, memorandums, notes, and data.

Reports (including QA reports) will be filed with correspondence. Analytical laboratory documentation, field data, and notes will be filed with the laboratory data. Filed materials may only be removed by authorized personnel on a temporary basis. The name of the person removing the file will be recorded. Laboratories will retain project files and data packages for 7 years, unless otherwise agreed.

26.3 Sample Identification Procedures

A sample numbering system will be used to identify each sample, including duplicate, field blank and equipment blank samples. The sample number will be a unique identifier.

Each sample, regardless of analytical protocol, will also be assigned a CH2M HILL site-specific identifier, which will contain a site- and sample-specific location identifier that indicates where the sample was obtained.

The sample number and station location identifier will be included on the sample tag, the traffic report, and the chain-of-custody record.

The site-specific identifier is based on the following system:

- **Site**—SRS (Southeast Rockford Site).
- **Station Location**—The station location identifier is the unique name of the sampling location (for example, soil gas "SG", pore water "PW", or surface water "SW"). Immediately following the sampling location type is the unique sampling location identifier that indicates exactly where the sample was collected. The first two numbers will represent the location. For example, the sample station location will be "SG-01" for a soil gas sample collected from location one.
- **QC Samples**—Field duplicates will have an "FD" appended to the end of the station location identifier. Field blanks will have a "FB" identifier and equipment blanks will have an "EB" identifier followed by the sample date. MS/MSDs are not identified in the station location identifier, but on the tag and the chain-of-custody form. QC samples will be sequentially numbered, and a record of those assigned will be kept in the field logbook and sample processing log.

26.3.1 Examples

- SRS-SG-01 is a soil gas (SG-XX) from location 1
- SRS-PW-01FD is a field duplicate of pore water sample SRS-PW-01
- SRS-FB-11302013 is a field blank collected on November 30, 2013.

26.4 Chain-of-Custody Procedures

Chains of custody will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information in accordance with the FOP. Sample information will include sample identification, date and time collected, number and type of containers, preservative information, analysis method, and comments. The chain of custody will also have the sampler's name and signature. The chain of custody will link location of the sample from the field logbook and sample processing log through sample disposal by the laboratory. See Appendix C for an example of a chain-of-custody form and sample tag. The laboratory will use the sample information to populate the laboratory database for each sample.

Worksheet #28-1—Analytical Quality Control and Corrective Action

Matrix: Soil Gas, Pore Water, Surface Water, IDW-Soil, IDW-Aqueous

Analytical Groups: VOCs

Concentration Level: Low

Sampling SOP: FOP #1, #2 and Appendix D

Analytical Method/ SOP Reference: Laboratory SOP #1 and Laboratory SOP #2

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Performance Check	Prior to IC, CC, and every 12 hours	Must meet method criteria.	Retune instrument and verify.	Laboratory analyst	
Method Blank	1/Extraction Batch	All analytes < RL	Correct problem, then re-prepare and analyze MB and all samples processed with the contaminated blank.	Laboratory analyst	Accuracy/Bias
Laboratory Duplicate	1/20 Samples	RPD < 30%	If the RPD is outside of either precision tolerances, flag duplicate results.	Laboratory analyst	Precision
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Internal Standard	Every sample, spiked sample, standard, and MB	Area within -50% to +100% of IC midpoint std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Laboratory analyst	Precision
Surrogate Spike	Every sample, spiked sample, standard, and MB	Recovery within limits stated in method	Correct the problem and reanalyze sample.	Laboratory analyst	Accuracy/Bias

IC = initial calibration, CC = continuing calibration, MB = method blank, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, RPD = relative percent difference, GC = gas chromatography

Worksheet #28-2—Analytical Quality Control and Corrective Action

Matrix: IDW-Soil, IDW-Aqueous

Analytical Groups: SVOCs

Concentration Level: Low

Sampling SOP: NA

Analytical Method/ SOP Reference: Laboratory SOP #5

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1/Extraction Batch	All analytes < RL	Correct problem, then re-prepare and analyze MB and all samples processed with the contaminated blank.	Laboratory analyst	Accuracy/Bias
MS/MSD	1/20 Samples	Recovery within limits stated in method and RPD < 30%	If the MS and/or MSD are outside of either accuracy or precision tolerances, flag MS/MSD results.	Laboratory analyst	Accuracy/Bias
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Internal Standard	Every sample, spiked sample, standard, and MB	Area within -50% to +100% of IC midpoint standard	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Laboratory analyst	Precision
Surrogate Spike	Every sample, spiked sample, standard, and MB	Recovery within limits stated in method	Correct the problem and reanalyze sample.	Laboratory analyst	Accuracy/Bias

MB = method blank, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, RPD = relative percent difference

Worksheet #28-3—Analytical Quality Control and Corrective Action

Matrix: IDW-Soil, IDW-Aqueous

Analytical Groups: Pesticides

Concentration Level: Low

Sampling SOP: NA

Analytical Method/ SOP Reference: Laboratory SOP #6

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1/Extraction Batch	All analytes < RL	Correct problem, then re-prepare and analyze MB and all samples processed with the contaminated blank.	Laboratory analyst	Accuracy/Bias
MS/MSD	1/20 Samples	Recovery within limits stated in method and RPD < 30%	If the MS and/or MSD are outside of either accuracy or precision tolerances, flag MS/MSD results.	Laboratory analyst	Accuracy/Bias
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Surrogate Spike	Every sample, spiked sample, standard, and MB	Recovery within limits stated in method	Correct the problem and reanalyze sample.	Laboratory analyst	Accuracy/Bias

MB = method blank, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, RPD = relative percent difference

Worksheet #28-4—Analytical Quality Control and Corrective Action

Matrix: IDW-Soil, IDW-Aqueous

Analytical Groups: Herbicides

Concentration Level: Low

Sampling SOP: NA

Analytical Method/ SOP Reference: Laboratory SOP #7

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1/Extraction Batch	All analytes < RL	Correct problem, then re-prepare and analyze MB and all samples processed with the contaminated blank.	Laboratory analyst	Accuracy/Bias
MS/MSD	1/20 Samples	Recovery within limits stated in method and RPD < 30%	If the MS and/or MSD are outside of either accuracy or precision tolerances, flag MS/MSD results.	Laboratory analyst	Accuracy/Bias
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Surrogate Spike	Every sample, spiked sample, standard, and MB	Recovery within limits stated in method	Correct the problem and reanalyze sample.	Laboratory analyst	Accuracy/Bias

MB = method blank, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, RPD = relative percent difference

Worksheet #28-5—Analytical Quality Control and Corrective Action

Matrix: IDW-Soil, IDW-Aqueous

Analytical Groups: PCBs

Concentration Level: Low

Sampling SOP: NA

Analytical Method/ SOP Reference: Laboratory SOP #8

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1/Extraction Batch	All analytes < RL	Correct problem, then re-prepare and analyze MB and all samples processed with the contaminated blank.	Laboratory analyst	Accuracy/Bias
MS/MSD	1/20 Samples	Recovery within limits stated in method and RPD < 30%	If the MS and/or MSD are outside of either accuracy or precision tolerances, flag MS/MSD results.	Laboratory analyst	Accuracy/Bias
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Surrogate Spike	Every sample, spiked sample, standard, and MB	Recovery within limits stated in method	Correct the problem and reanalyze sample.	Laboratory analyst	Accuracy/Bias

MB = method blank, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, RPD = relative percent difference

Worksheet #28-6—Analytical Quality Control and Corrective Action

Matrix: Pore Water, Surface Water, IDW-Soil, IDW-Aqueous

Analytical Groups: Hardness and Metals (including mercury)

Concentration Level: Low

Sampling SOP: Appendix D

Analytical Method/ SOP Reference: Laboratory SOPs #4 and #9

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Preparation Blank	1/Analytical Batch	All analytes < RL	Reanalyze preparation blank. If fails, analyze a calibration blank. Re-prepare/reanalyze analytical batch as appropriate.	Laboratory analyst	Accuracy/Bias
Serial Dilution (ICP Analysis Only)	1/20 Samples	Concentration of reported analytes are > 50 times the MDL and RPD > 10%.	Correct problem, then re-prepare and analyze the serial dilution and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Internal Standards (ICP Analysis Only)	Every sample, spiked sample, standard, and preparation blank	Recovery within limits stated in method.	Inspect mass spectrometer for malfunctions; dilute the sample and reanalyze.	Laboratory analyst	Precision
MS/MSD	1/20 Samples	Recovery within limits stated in method and RPD < 30%.	If the MS and/or MSD are outside of either accuracy or precision tolerances, flag MS/MSD results.	Laboratory analyst	Accuracy/Bias
Post-digestion Spike (PDS)	After any analyte (except Ag and Hg) fails spike %R	Recovery within limits stated in method.	If the PDS is outside of accuracy tolerances, flag PDS results.	Laboratory analyst	Accuracy/Bias
Laboratory Duplicate	1/Analytical Batch	Concentration of reported analytes are > 5 times the reporting limit and RPD > 20%.	If the duplicate is outside of accuracy tolerances, flag duplicate results.	Laboratory analyst	Accuracy/Bias
LCS	1/Analytical Batch	Recovery within limits stated in method.	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity

ICP = inductively coupled plasma, MS/MSD = matrix spike/matrix spike duplicate, LCS = laboratory control sample, PDS = post digestion spike, RPD = relative percent difference

Worksheet #28-7—Analytical Quality Control and Corrective Action

Matrix: IDW-Soil, IDW-Aqueous

Analytical Groups: pH and Flashpoint

Concentration Level: Low

Sampling SOP: NA

Analytical Method/ SOP Reference: Laboratory SOP #10 and #11

Sampler's Name: Field Team Member

Field Sampling Organization: CH2M HILL

Analytical Organization: TestAmerica, Inc.

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
LCS	1/Extraction Batch	Recovery within limits stated in method	Correct problem, then re-prepare and analyze the LCS and all samples in the affected analytical batch.	Laboratory analyst	Sensitivity
Laboratory Duplicate	1/Analytical Batch	Concentration of reported analytes are > 5 times the reporting limit and RPD > 20%.	If the duplicate is outside of accuracy tolerances, flag duplicate results.	Laboratory analyst	Accuracy/Bias

LCS = laboratory control sample, RPD = relative percent difference

Worksheet #29—Project Documents and Records

Sample Collection Documents and Records	Onsite Analysis Documents and Records	Offsite Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes Boring Logs Scribe Database Chain-of-Custody Records Air Bills Custody Seals Telephone Logs Corrective Action Forms	Sample Receipt, Custody, and Tracking Records Equipment Calibration Logs Equipment Maintenance, Testing, and Inspection Logs Corrective Action Forms Reported Field Sample Results Telephone Logs Analysis Logs	Sample Receipt, Custody, and Tracking Records Narrative Standard Traceability Logs Equipment Calibration Logs Sample Preparation Logs Run Logs Equipment Maintenance, Testing, and Inspection Logs Corrective Action Forms Reported Field Sample Results Reported Results for Standards, QC Checks, and QC Samples Instrument Printout (raw data) for Field Samples, Standards, QC Checks, and QC Samples Data Package Completeness Checklists Extraction/Cleanup Records Raw Data (stored electronically) QA Review Records	Data Verification/Validation Reports Corrective Action Forms	Project database for analytical and field data Health and saftey briefing information Staff health and safety records

Electronic analytical data will be similar in format to that of the CLP laboratory. The laboratory data package will be organized such that the analytical results are reported on a per-analytical-batch basis, unless otherwise specified. In addition to the summary data deliverable, a full-supporting raw data deliverable package is required from the laboratory. All data will be provided electronically as a PDF file. CH2M HILL will provide data copies to USEPA.

An EDD is also required for all data. The laboratory will provide CH2M HILL with an EDD in the current EQuIS format. The data will undergo QA reviews prior to being loaded to the project database. Delivery time for data from the laboratory will vary based on project-specific data use.

29.1 Sample Tracking Program

The USEPA Scribe program will be used for field documentation and generation of chains of custody. Refer to USEPA Office of Solid Waste and Emergency Response 9240.0-44, EPA 540-R-07-06 Contract Laboratory Program Guidance for Field Samplers, dated July 2007.

29.2 Project-specific Deliverables

Analytical data will be exported into a format consistent with the EDD format specified by USEPA Region 5. Data will be submitted to USEPA Region 5 in accordance with the requirements located here: http://www.epa.gov/region5superfund/edman/. Documentation and reports specified in this QAPP will be retained in Adobe PDF format.

Worksheets #31, #32, and #33—Assessments and Correction Action

Assessments:

Assessment Type	Frequency	Assessment Deliverable	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)	Projected Delivery Date(s)
Field progress report	Daily	Summarizing e-mail	Dave Shekoski, field team leader, CH2M HILL	Renee Hunt, site manager, CH2M HILL	Daily during field activities
Data review and verification	For all analytical delivery packages	E-mail of deficiencies	Shannon Olson, project chemist, CH2M HILL	TestAmerica, Inc., QA officer	After arrival of data from the laboratory and during data verification activities
Data validation	One after all data are validated	Data validation report	Shannon Olson, project chemist, CH2M HILL	Renee Hunt, site manager, CH2M HILL	21 business days after receipt of validated data
Data evaluation	One after project completion	Soil Gas Technical Memorandum	Renee Hunt, site manager, CH2M HILL	Timothy Drexler, work assignment manager, USEPA	45 business days after receipt of validated data
Data evaluation	One after project completion	Pore Water Technical Memorandum	Renee Hunt, site manager, CH2M HILL	Timothy Drexler, work assignment manager, USEPA	45 business days after receipt of validated data

Assessments and Corrective Action:

Assessment Type	Corrective Action Documentation	Person(s) Responsible for Identifying and Implementing Corrective Actions (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of Corrective Action (Title and Organizational Affiliation)	Timeframe for Corrective Action
Daily field documentation reviews	Field notes/ e-mail	Dave Shekoski, field team leader, CH2M HILL	Renee Hunt , site manager, CH2M HILL Dave Shekoski, field team leader, CH2M HILL	As soon as notification of corrective action is received
Data review and verification	Corrective action reports and/or updated case narratives and corrected data submissions	Shannon Olson, project chemist, CH2M HILL TestAmerica, Inc., QA officer	Shannon Olson, project chemist, CH2M HILL	3 to 5 business days

Worksheet #34—Data Verification and Validation Inputs

To ensure that scientifically sound data of known and documented quality are used in making environmental decisions, the following three-step data review will be performed. Step I (verification) will confirm that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Step II (validation) will assess whether the sampling and analytical processes comply with the contract-specific and the QAPP-specific requirements. Step III (usability assessment) will determine whether the resulting data are suitable as a basis for the decision being made. Worksheets #34 to #37 describe the processes to be followed. Worksheet #34 establishes the procedures that will be followed to verify project data including, but not limited to, sampling documents and analytical data package. The items subject to verification and validation are listed in the following table.

Item	Description	Verification (completeness)	Validation (conformance to specifications)
		Planning Documents/Records	
1	Approved QAPP	X	
2	Field SOPs	X	
3	Laboratory SOPs	X	
		Field Records	
4	Field logbooks	X	X
5	Sample processing logs	X	X
6	Boring logs	X	X
7	Chain-of-Custody Forms	X	X
8	Field corrective action reports	X	X
		Analytical Data Package	
9	Cover sheet (laboratory identifying information)	X	X
10	Case narrative	X	X
11	Internal laboratory chain-of-custody	X	X
12	Sample receipt records	X	X
13	Sample chronology (that is, dates and times of receipt, preparation, and analysis)	X	X
14	RL/MDL establishment and verification	X	X
15	Standards traceability	X	X
16	Instrument calibration records	X	X
17	Definition of laboratory qualifiers	X	X
18	Results reporting forms	X	X
19	QC sample results	X	X
20	Corrective action reports	X	X
21	Electronic data deliverable	X	X

Worksheet #35—Data Verification Procedures

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Field Notes	Verify that records are present and complete for each day of field activities. Verify boring logs. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements.	Internal	Dave Shekoski, field team leader, CH2M HILL Renee Hunt, site manager, CH2M HILL
Chain-of-Custody and Shipping Forms	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook and sample processing log. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (for example, MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Internal/ External	Dave Shekoski, field team leader, CH2M HILL Shannon Olson, project chemist, CH2M HILL TestAmerica, Inc. , QA Manager
Laboratory Data	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the chain-of-custody's to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Internal	Shannon Olson, project chemist, CH2M HILL

Worksheet #36—Data Validation Procedures

Matrix	Analytical Group	Required Deliverable	Validation Percentage	Validation Criteria	Data Validator (title and organizational affiliation)
Soil Gas	Select VOCs	Level IV Data Report and EQuIS-compatible EDD	100% Level III and 10% Level IV	Manual Stage 2A[a] and Stage 2B[a] validation per USEPA National Functional Guidelines, laboratory SOPs, and QAPP criteria	Shannon Olson, project chemist, CH2M HILL
Pore Water and Surface Water	Select VOCs and Hardness	Level IV Data Report and EQuIS-compatible EDD	100% Level III and 10% Level IV	Manual Stage 2A[a] and Stage 2B[a] validation per USEPA National Functional Guidelines, laboratory SOPs, and QAPP criteria	Shannon Olson, project chemist, CH2M HILL

[a] Stage 2A and Stage 2B per, "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (Office of Solid Waste and Emergency Response No. 9200.1-85, EPA 540-R-08-005, January 13, 2009)

36.1 Data Verification/Validation Scope Overview

The CH2M HILL project chemist will perform a Stage 2A and Stage 2B data validation in accordance with the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (USEPA 2009), the site-specific QAPP, and laboratory SOPs on 100 percent of the laboratory-generated data from TestAmerica, Inc. USEPA's National Functional Guidelines will be used as guidance for the data validation.

36.2 Field Data Review

Field-generated information may include field logbooks, boring logs, sample chain-of-custody forms, shipping documents, sampling observations, sample labels, and other miscellaneous field observations. All field measurements and or field log information will be entered into field logbooks and reviewed daily by the field team leader or designee. The designee may be a qualified field geologist, engineer, environmental scientist, and/or technician.

36.3 Laboratory Data Review Requirements

All analytical data generated by the laboratory will be verified before submittal to the CH2M HILL project chemist. The internal data review process, which is multitiered, will include all aspects of data generation, reduction, and QC assessment. In each laboratory analytical section, the analyst performing the tests shall review 100 percent of the definitive data. After the analyst's review has been completed, 100 percent of the data will be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria.

Elements for review or verification at each level must include, but not be restricted to, the following:

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate SOPs and analytical methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations
- Appropriate application of QC samples and compliance with established control limits

- Documentation completeness (for example, all anomalies in the preparation and analysis have been identified, appropriate corrective actions have been taken and documented in the case narrative[s], associated data have been appropriately qualified, and anomaly forms are complete)
- Accuracy and completeness of data deliverables (electronic)

36.4 Laboratory Data Evaluation

The calibration, QC, corrective actions, and flagging requirements for definitive data are shown in Worksheets #12-1, #12-2, #12-3, #15-1, #15-2, #15-3, #24, #28-1, #28-2, #28-3, #28-4, #28-5, #28-6, and #28-7. The laboratory may apply data qualifiers based on their review or add a note in the laboratory case narrative. The definitions of any data qualifiers applied by the laboratory must be defined in the case narrative. The data qualifiers are reviewed by the supervisor of the respective analytical sections after the first and second level reviews of the laboratory data have been performed.

36.5 Data Review Guidelines

The laboratory assessment of the data quality will be reviewed for completeness and accuracy. Data review will be done manually and will include, but is not limited to, the following:

- Sampling documentation (such as the chain-of-custody form)
- Preservation summary and technical holding times
- Presence of all analyses and analytes requested
- Use of the required sample preparation and analysis procedures
- The method detection and reporting limits will be evaluated against the project requirements
- The correctness of the concentration units
- Case narrative

36.6 Data Verification/Validation Guidelines

The data verification process builds on data review. Project data will be reviewed and verified as part of the data assessment for this project. The review will be performed on an analytical batch basis by assessing QC samples and associated field sample results. Data verification guidelines have been developed in accordance with the method requirements, professional judgment, and general USEPA National Functional Guidelines.

Summary data review and verification will be performed as follows:

- Chain-of-custody documentation
- Holding time
- QC sample frequencies
- Method and field blanks
- Laboratory control samples
- Surrogate spikes
- MS/MSD
- Initial and continuing calibration information
- Field duplicate precision
- Case narrative review and other method-specific criteria

The manual verification/validation process includes data flagging for issues related to method blanks, laboratory control samples, MS/MSD samples, field duplicates, surrogate recoveries, holding time, and reconciliation of dilutions and re-extractions. Data flags, as well as the reason for each flag, are entered into an electronic database and made available to the data users. A final flag is applied to the data by the CH2M HILL project chemist after evaluating all flags entered into the database and selecting the most conservative of the verification flags.

If, during the data review and verification process, a systematic problem or other major issue with the data is identified, the CH2M HILL project chemist will contact the laboratory's project manager or QA manager. Additional evaluation of the data may be performed including an in-depth review of the raw data to verify accuracy followed by analysis and interpretation of the data in the context of the project objectives and end-use as part of the usability assessment.

A data validation report will be prepared summarizing the findings and discussing their impact on the overall data usability. It will be incorporated into the final data evaluation summary report.

36.7 Flagging Conventions

CH2M HILL-specific final data qualifier definitions are summarized in Table 36-1.

TABLE 36-1
Verification/Validation Data Qualifiers

Qualifier	Description
R	The data are rejected because of deficiencies in meeting QC criteria and may not be used for decision making.
J	Estimated: The analyte was positively identified; the quantitation is an estimation because of discrepancies in meeting certain analyte-specific QC criteria.
UJ	The analyte was not detected; however, the result is estimated because of discrepancies in meeting certain analyte-specific QC criteria.
UB	Undetected due to blank contamination. The analyte was detected in the sample and in an associated method, field, or trip blank. The quantity of the analyte is deemed undetected because it falls below the 95 percent confidence interval (five times the blank concentration). The analyte concentration is potentially the result of contamination.
U	Undetected: The analyte was analyzed for, but not detected or is qualified as nondetect because of blank contamination.

Table 36-2 presents the general data validation guidelines with consideration to the USEPA National Functional Guidelines to be used by CH2M HILL for applying the data qualifiers.

TABLE 36-2
Data Qualifying Conventions—General

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis by a factor of 2 or more	J for positive results; R or UJ for nondetects*	All analytes in the sample
Sample Preservation	Sample not preserved (if sample preservation was not done in the field but was performed at the laboratory upon sample receipt, no flagging is required)	J for positive results; R or UJ for nondetects*	Sample
	Temperature out of control	J for positive results; UJ for nondetects*; R based on professional judgment	Sample
Sample Integrity (SW8260)	Bubbles in VOA vial greater than 4 millimeters (pea size) used for analysis	J for the positive results; UJ for nondetects; R based on professional judgment	Sample
Instrument Tuning	Mass assignment error or Ion abundance method-specific criteria not met	R for all results, if critical ions involved, use judgment otherwise	All associated samples in analytical batch
Initial Calibration	All analytes must be within method specified criteria	J for positive results; UJ for nondetects; R based on professional judgment	All associated samples in analytical batch
Second Source Check or Continuing Calibration	All analytes must be within method specified criteria	High Bias: J for positive results, no flag for nondetects Low Bias: J for positive results, UJ for nondetects J positive/R all nondetects greater than twice the control criteria	All associated samples in analytical batch
Low-level Calibration Check or Interference Check Sample (SW6010 Only)	All analytes must be within 20% of expected value	High Bias: J positive results, no flag for nondetects Low Bias: J positive results, UJ nondetects J positive/R all nondetects greater than twice the control criteria	All associated samples in analytical batch
Internal Standards	Area greater than UCL Area less than LCL Sample is re-extracted and reanalyzed and recovery outside of criteria is confirmed as a matrix effect	J for positive results J for the positive results; UJ for the nondetects If area is too low based on professional judgment, UJ or R nondetects	Sample
Surrogates	%R greater than UCL %R less than LCL and greater than 10% %R less than 10% Excessive dilution	J for positive results J for positive results; UJ for nondetects J for positive results; R for nondetects No flag required	Sample
LCS	%R greater than UCL	J for the positive results;	The specific analyte(s) in all samples

TABLE 36-2
Data Qualifying Conventions—General

QC Requirement	Criteria	Flag	Flag Applied To
	%R less than LCL and greater than 10% %R less than LCL and less than 10%	J for the positive results; UJ for the nondetects J for the positive results; R for the nondetects	in the associated analytical batch
Blanks (Method, Field, Equipment, Trip or Calibration)	Analyte(s) detected (use the blank of the highest concentration)	UB for positive sample results ≤ 5x highest blank concentration (10x for common laboratory contaminants)	All samples in preparation, field or analytical batch, whichever one applies
Field duplicates or Laboratory Duplicates	RPD > 30% RPD > 20%	J for positive results, no flag for nondetects	The specific analyte(s) in the associated sample Note: No flagging is required for results less than the reporting limit
MS/MSD and Post-digestion Spikes	%R greater than UCL %R less than LCL and >10% %R less than 10% or MS/MSD RPD > 30% Sample concentration > 4 times the spike concentration Excessive dilution*	J for positive results J for positive results; UJ for nondetects J for positive results; R for nondetects J for positive results No flag required No flag required	The specific analyte(s) in the parent sample
Serial Dilution (SW6010 Only)	All analytes must be within 10% of expected value Sample concentration < 50× MDL	J positive results, UJ nondetects No flag required	The specific analyte(s) in the parent sample
Retention Time Window	Analyte within established window	R for all results	Sample
Canister Pressure (not applicable to Grab Samples) Initial vacuum should be 28 inches Hg (30 inches fully evacuated)	If Initial pressure 24 to 26 inches Hg	J for positive results; UJ for nondetects	Sample
	If < 24 inches of Hg—Reject all data if used	R for all results	Sample
	If final pressure >10 <20 inches Hg—sampling time may be extended if DQOs allow or the laboratory will dilute the sample	No Flag required	Sample
Final vacuum should be between 2 and 10 inches Hg (5 inches ideal)	If Final pressure > 20 inches Hg: Slight change (20 to 27 inches)	J for positive results; UJ for nondetects	Sample
	No change in pressure	Apply R to all data	Sample

TABLE 36-2
Data Qualifying Conventions—General

QC Requirement	Criteria	Flag	Flag Applied To
Canister/Flow controller Certification (Individual)	Analyte(s) detected greater than RL	UB for positive sample results ≤ 5x blank concentration (10× for common laboratory contaminants)	Sample

* Based on analyte-specific review

%R = percent recovery, LCL = lower confidence limit, LCS = laboratory control sample, MS = matrix spike, MSD = matrix spike duplicate, UCL = upper confidence limit

Worksheet #37—Data Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following subsections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision making for the project, and describes how data quality issues will be addressed and how limitations of the use of the data will be handled.

37.1 Summary of Usability Assessment Processes

It is the responsibility of the CH2M HILL project chemist and the laboratory to ensure that the data meet the detection limits and laboratory QC limits listed in this QAPP. During the data verification assessment, non-conformances are documented and data are qualified for use in decision making. The data are determined to be usable by the project chemist based on the requirements of this QAPP. Data gaps will be present if a sample is not collected, a sample is not analyzed for the requested parameters, or the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis. All data are usable as qualified by the data validator, with the exception of rejected data. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis. There is no generic formula for determining whether a result is an outlier. Potential outliers will be referred to a statistician and senior consultant, who will determine which formulas are appropriate for classifying data points in a statistically appropriate and defendable manner.

37.2 Evaluation Procedures to Assess Project-specific Overall Measurement Error

In-depth assessment occurs during the data verification process. The verification will assess conformance with the requirements of the methods, SOPs, and objectives of this QAPP. The findings of the data verification process will generate qualifiers applied to the data considered in context to assess overall usability of the data.

37.3 Personnel Responsible for Performing Usability Assessment

- Shannon Olson, project chemist, CH2M HILL
- Kari MacGregor, database manager, Critigen
- Frank Dillon, senior technical consultant, CH2M HILL
- Tiffany Swoveland Chapman , project delivery lead, CH2M HILL
- Daniel Lavoie, pore water subject matter expert, CH2M HILL
- Barrie Selcoe, soil gas subject matter expert, CH2M HILL
- Jennifer Simms, soil gas technical manager, CH2M HILL
- Renee Hunt, site manager, CH2M HILL
- Dave Shekoski, field team leader, CH2M HILL

37.4 Usability Assessment Documentation

The data verification report will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field duplicates. All the results will be

assembled and statistically reported for an overall quality assessment provided in the final data evaluation summary report. Discussion will cover precision, accuracy, representativeness, comparability, and completeness defined as follows.

37.4.1 Precision

Laboratory precision is measured by the variability associated with duplicate (two) or replicate (more than two) analyses.

Total precision is the measurement of the variability associated with the entire sampling and analytical process. It is determined by analysis of duplicate field and/or laboratory samples and measures variability introduced by both the laboratory and field operations. Field duplicate and MS/MSD samples will be analyzed to assess field and laboratory precision. For duplicate sample results, the precision is evaluated using the RPD. For replicate results, the precision is measured using the relative standard deviation (RSD). The formula for the calculation of RPD is provided below.

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2)\ x\,100\%}{(C_1 + C_2)\ /\ 2} \qquad (1)$$

Where:

RPD = relative percent difference
C_1 = larger of the two observed values
C_2 = smaller of the two observed values

If calculated from three or more replicates, use RSD rather than RPD:

$$RSD = (s\ /\ \overline{y})\ x\,100\% \qquad (2)$$

Where:

RSD = relative standard deviation
s = standard deviation
$\overline{y}$ = mean of replicate analyses

Standard deviation, s, is defined as follows:

$$S = \sqrt{\sum_{i=1}^{n} \frac{(yi - \overline{y})^2}{n-1}} \qquad (3)$$

Where:

S = standard deviation
yi = measured value of the ith replicate
$\overline{y}$ = mean of replicate analyses
n = number of replicates

37.4.2 Accuracy

Accuracy reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into

an LCS and/or MS/MSD to a control limit. Surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements.

The formula for calculation of accuracy is included below as percent recovery (%R).

$$\%R = 100\%\ x \left[\frac{S - U}{C_{sa}} \right] \tag{4}$$

Where:

%R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

For situations where a standard reference material is used instead of or in addition to MSs:

$$\%R = 100\%\ x \left[\frac{C_m}{C_{sm}} \right] \tag{5}$$

Where:

%R = percent recovery
C_m = measured concentration of standard reference material
C_{sm} = actual concentration of standard reference material

37.4.3 Representativeness

Representativeness is the degree to which sample data accurately reflect the characteristics of a population of samples. It is achieved through a well-designed sampling program and by using standardized sampling strategies and techniques and analytical procedures. Factors that can affect representativeness include sample collection, storage, preservation procedures, site homogeneity, sample homogeneity at a single point, and available information around which the sampling program is designed.

37.4.4 Completeness (Statistical)

Completeness is a measure of the amount of valid data obtained compared with the amount expected under correct, normal conditions. It is calculated for the aggregation of data for each analyte measured as a compound of concern for the project objectives. Valid data are data that are usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed. The goal for completeness, based on specific project goals, is 90 percent.

Defined as follows for all measurements:

$$\%C = 100\%\ x \left[\frac{V}{T} \right] \tag{6}$$

Where:

%C	=	percent completeness
V	=	number of measurements judged valid
T	=	total number of measurements

37.4.5 Comparability

Comparability is the confidence with which one data set can be compared to another. It is achieved by maintaining standard techniques and procedures for collecting and analyzing samples and reporting the analytical results in standard units.

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Figures

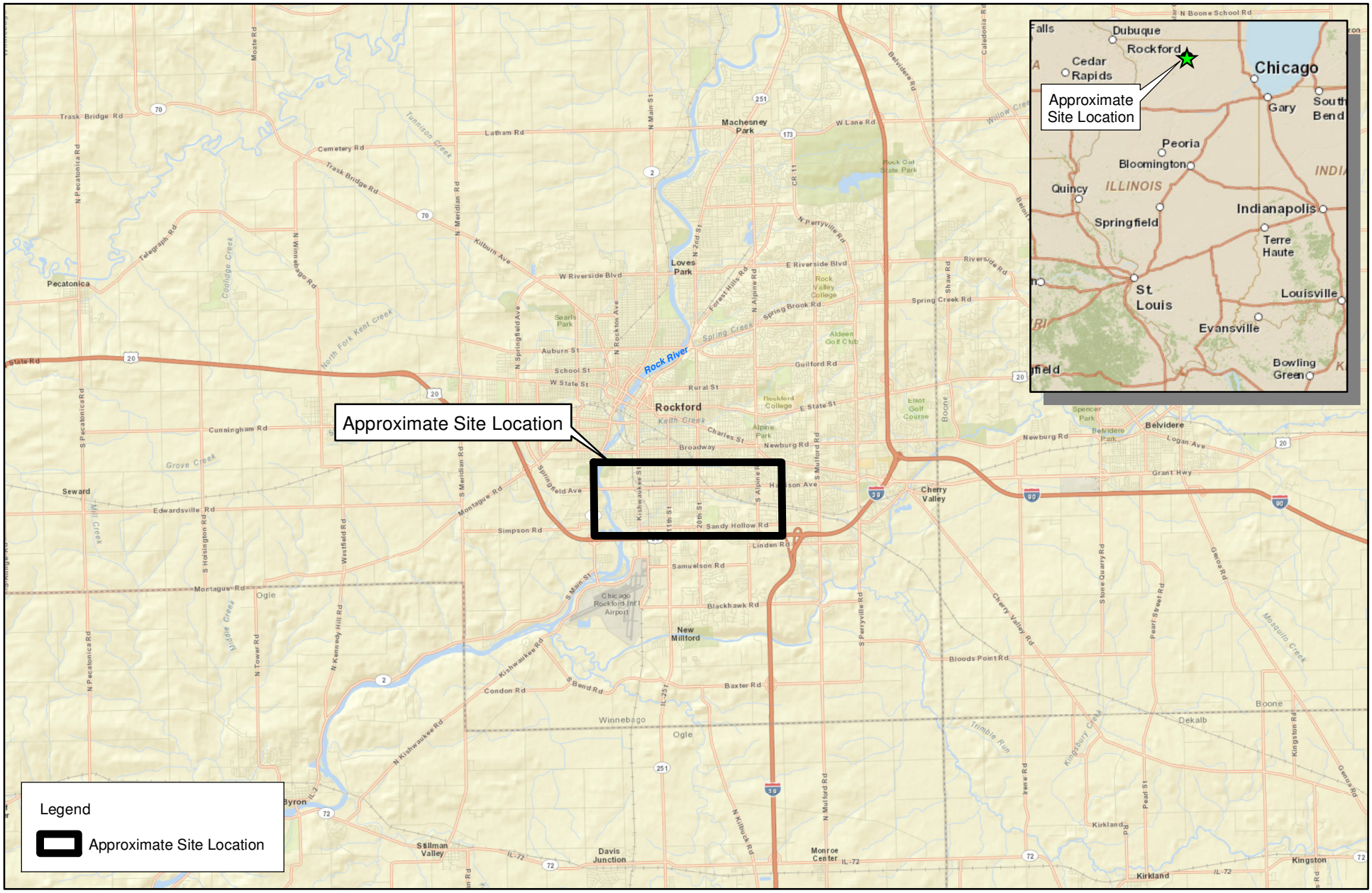

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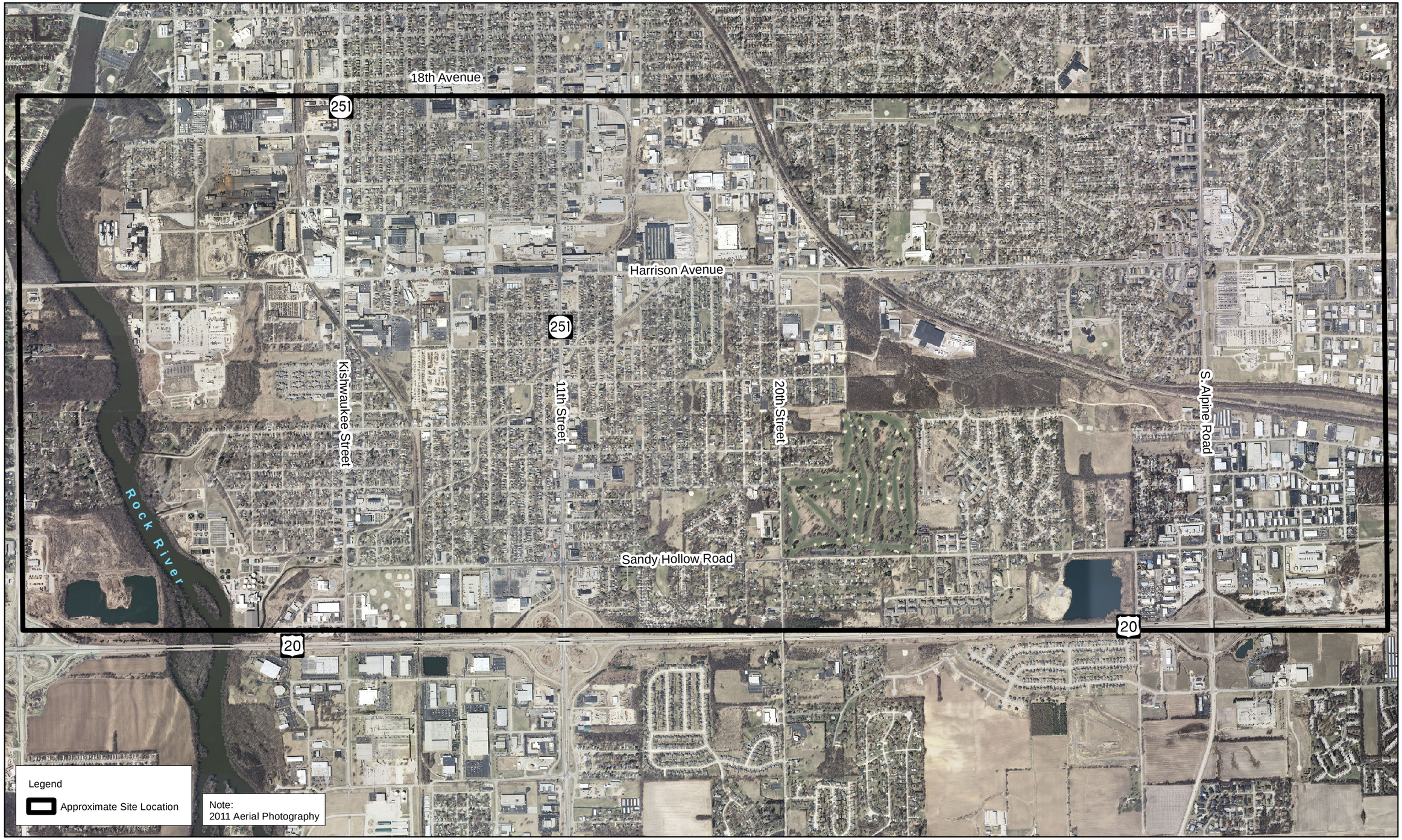
Figure 1
Site Map
Southeast Rockford Ground Water Contamination Site
Rockford, IL

CH2MHILL

E:\EPA\476243SOUTHEASTROCKFORD\MAPFILES\QAPP\FIGURE 01 - SITE MAP.MXD JHANSEN1 10/24/2013 2:34:54 PM



N

0 1,600 3,200

Feet

Figure 2
Site Area
Southeast Rockford Ground Water Contamination Site
Rockford, IL

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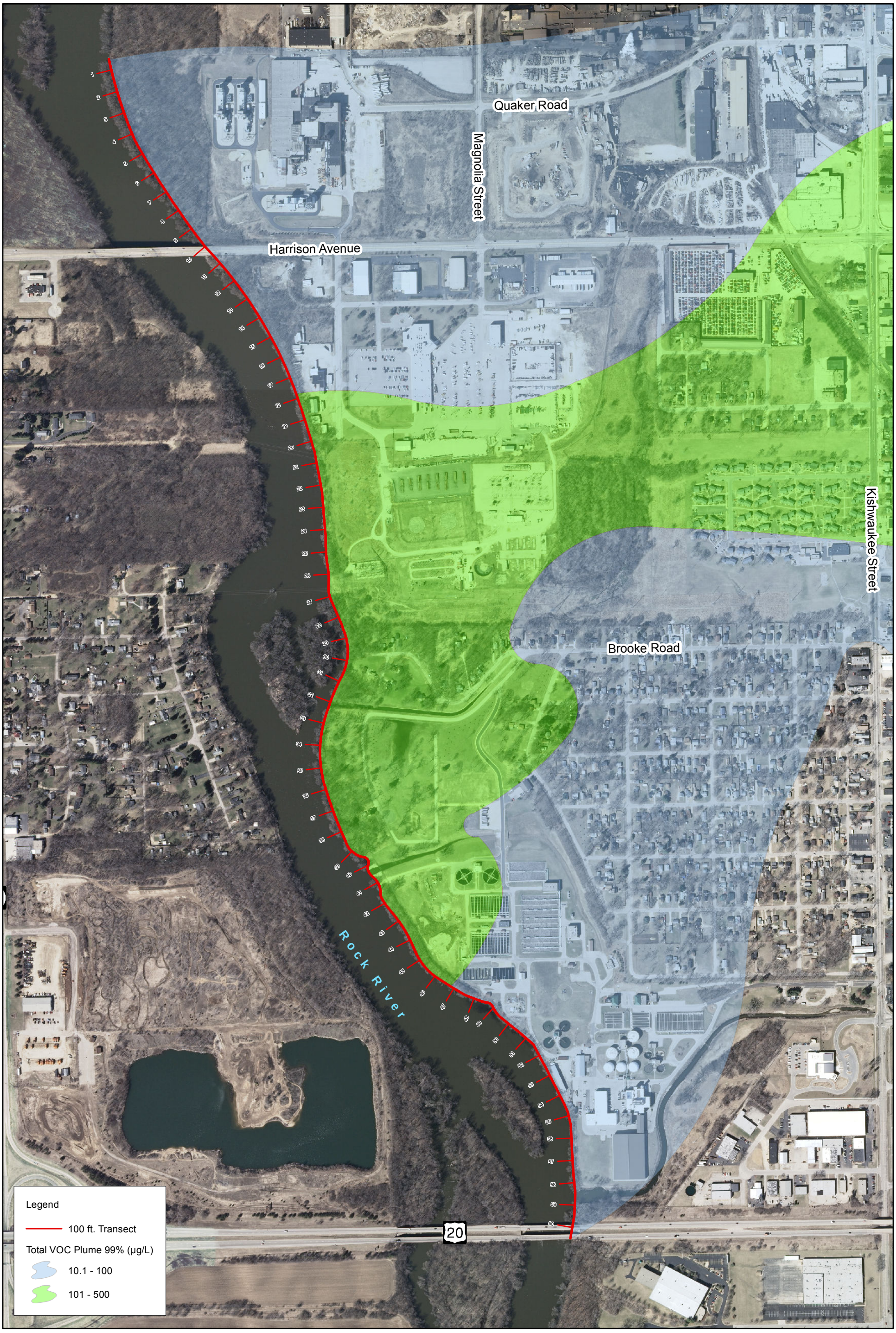
E:\EPA\476243SOUTHEASTROCKFORD\MAPFILES\QAPP\FIGURE 02 - SITE AREA.MXD JHANSEN1 10/25/2013 11:11:58 AM



0 600 1,200
Feet

Figure 3
Pore Water Locations
Southeast Rockford Ground Water Contamination Site
Rockford, IL

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E:\EPA\476243SOUTHEASTROCKFORD\MAPFILES\QAPP\FIGURE 03 - PORE WATER SAMPLING.MXD JHANSEN1 10/25/2013 4:32:23 PM

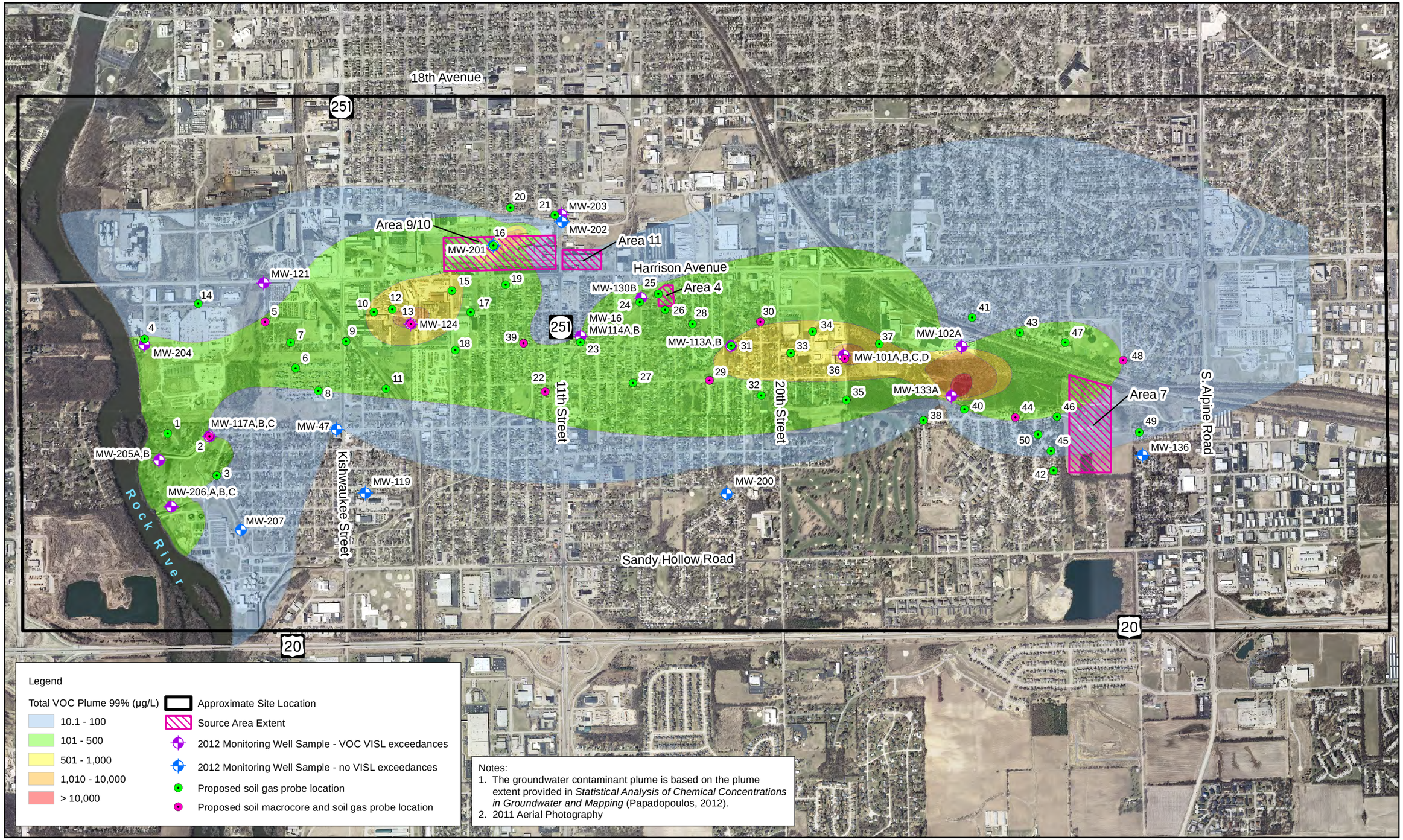

Figure 4
Soil Gas Probe Sampling
Southeast Rockford Ground Water Contamination Site
Rockford, IL

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E:\EPA\476243SOUTHEASTROCKFORD\MAPFILES\QAPP\FIGURE 04 - SOIL GAS PROBE SAMPLING.MXD JHANSEN1 12/10/2013 11:37:57 AM

Appendix A
Field Operating Procedures

FIELD OPERATING PROCEDURE 1

Installation of Permanent Soil Gas Sampling Probes

Purpose and Scope

This field operating procedure (FOP)is recommended as a practical approach for the installation of permanent soil gas sampling probes where the intent is to collect soil gas samples on multiple occasions. This FOP is commonly used during vapor intrusion assessments associated with subsurface volatile organic compound contamination. This FOP should be used when its application is consistent with the project's data quality objectives (DQOs) and in conjunction with FOP 2, *Collection of Soil Gas Samples from Temporary and Permanent Probes Using Canisters*. The project team is responsible for ensuring the procedure meets all applicable regulatory standards and receives approval/concurrence from the leading regulatory agency for the project. Only persons trained in the installation of soil gas probes should attempt this procedure.

Equipment and Materials

Materials and equipment vary depending on type of sampling. Typical equipment required includes the following:

- Geoprobe Systems soil gas implant method (The equipment listed below is typically supplied by the drilling subcontractor.):
  - Drive rods—1.25-inch-outer-diameter (OD) drive rods
  - Expendable drive points—steel or aluminum 1.1-inch-OD expendable drive points
  - Stainless steel probe screen, such as the Geoprobe Systems implants. Several screen lengths are available (6-inch, 14-inch, 21-inch), but for discrete intervals required in vapor intrusion investigations, a 6-inch screen is typically recommended.
  - Materials to create a flush-mount or stick-up casing
- AMS dedicated gas vapor probe method (The equipment may be rented or purchased from AMS; the company offers several different "Gas Vapor Probe Kits" that may be customized with additional pieces of equipment.):
  - Slide hammer or hammer drill for driving the probe (will need power supply for the hammer drill)
  - Drive rods—5/8-inch-OD hollow drive rods
  - Removal jack
  - Dedicated gas vapor probe tip assembly
  - Materials to create a flush-mount or stick-up casing
  - Leather work gloves, vice grips, and a large adjustable wrench are necessary for the AMS method
- Probe tubing—1/4-inch-OD Teflon tubing (may be supplied by the drilling subcontractor)
- Probe cap (to seal the tubing during equilibration)—Swagelok part number SS-400-C
- Glass beads (very small beads, similar to sand) to create a permeable layer around the probe screen.
- Bentonite seal mixture (25 percent glass beads and 75 percent powdered bentonite clay [high-yield type]) to grout the hole from above the screen to the ground surface.
- Electrical tape

- MultiRae five-gas meter for health and safety monitoring during drilling. A MultiRae photoionization detector and Landtec GEM 2000 landfill gas meter (or equivalent) may be used instead of the five-gas meter.

Procedures/Guidelines

1. As with all intrusive site work, a utility clearance should be performed prior to mobilization. It may also be necessary to acquire permits and site access.
2. Soil gas sampling should not be performed until 48 hours after a significant rain event (defined as greater than 1 inch of rainfall).
3. It is common practice to install soil gas probes using a drill rig (for instance, Geoprobe permanent soil gas implant method). Operation of such machinery shall be performed only by trained and licensed personnel. Soil gas probes can also be advanced with a hand tool method (such as the AMS Retract-A-Tip system). The hand tool installation method is only applicable to relatively shallow sampling (that is, up to 10 to 15 feet below ground surface [bgs] depending on the soil type).
4. The probe can be finished at the ground surface with a stick-up or flush-mount cover similar to groundwater monitoring wells.
5. Prior to attempting installing soil gas probes, there should be an understanding of the following subsurface conditions at the site:
   - 5.1. Depth to groundwater—Soil gas samples must be collected in the vadose zone (and above the capillary fringe).
   - 5.2. Soil permeability—It may not be feasible to collect soil gas from finer-grained or tight soils with little pore volume, such as clays. If clay layers are present in the subsurface, the intervals should be avoided. For sampling in soils, using permanent soil gas implants with a wider borehole is recommended. Care should be taken during purging and sampling so that the vacuum in the sampling system never exceeds 7 inches mercury, or approximately 100 inches water.
   - 5.3. Gravel or dense clay layers may make the hand tool installation method impracticable.
6. Select the probe interval length—Typically, probe sample intervals are 6 inches or 1 foot; however, smaller or larger intervals may be selected depending on the project's DQOs.
7. Select the probe depth interval, as follows:
   - 7.1. The top of the soil gas probe should be at least 5 feet bgs to avoid short circuiting with outdoor air. If there is impermeable ground cover (such as concrete or asphalt), shallower sampling depths may be considered.
   - 7.2. The bottom of the soil gas probe must be above the capillary fringe.
   - 7.3. As the depth of the sampling interval increases, the difficulty of installing the probe also increases, with the primary challenges being pushing the rods down with a hand tool.
   - 7.4. It is advisable to collect a soil core from the proposed sampling area prior to installing the soil gas probe to identify the exact depth of the capillary fringe and determine where the most permeable soil layers are located.
     - 7.4.1. Soil cores will be processed placing the cores on a decontaminated table (or other stable surface) and slitting a section of the liner sufficient to examine and remove the sample. Care will be taken to scrape away the smear zone on the skin of the core sample in order to inspect the structure of the sample. The cores will be visually characterized for soil type, particle size, color, moisture, consistency, and other visual observations (such as staining, odor). The soil will be described using visual identification based on the Unified Soil

Classification System in accordance with ASTM International D2488 standard practice. Digital photographs of each core will be taken to document the undisturbed core structure. Each photograph will include a scale (for example, tape measure), station identification, core interval, and date of core collection. A standard core log will be used to record all the observations (Attachment 1).

7.5. Sampling at multiple depths at each sample location (or a subset of the locations) should be considered to obtain a vertical profile of soil gas conditions if the vadose zone height is long enough; typically, the bottom of a probe should be at least 5 feet from the top of the probe beneath it. Multi-depth probes can be installed in one hole by starting with the deepest depth first and then continuing upward.

Permanent Soil Gas Probe System Setup

1. Obtain soil gas sampling probes in sufficient quantity to carry out the assessment. See Figure 1. The systems and their installation can be obtained from geotechnical firms that provide direct-push supplies and services or from AMS for the hand tool method. Their basic installation procedures can be followed as long as the details below are included.
2. Manufactured soil gas probes (such as the Geoprobe Soil Gas Implant, and AMS dedicated gas vapor probe tip) are specifically manufactured for soil gas collection and facilitate installation, improve sampling, are easily decontaminated between each use, and offer consistency and ease of use.
3. It is necessary to coordinate the hardware (that is, size of tubing, fittings, sampling interface assembly, etc.) that mates the soil gas probe sampling line to the sampling system (such as, Tedlar bags, SUMMA canisters). Appropriate hardware is critical to achieving a leak-free system. All connections should be inert gas-tight compression fittings (Swagelok or equivalent), and all sample transfer lines should be made of Teflon or tubing. Typically, all tubing and fittings should be 1/4-inch-OD. The fittings will match up with the sampling manifold specified in the soil gas sampling FOP.
4. The soil gas probes and equipment must be decontaminated prior to use. Steam cleaning is the preferred method of decontamination; however, a three-stage decontamination process consisting of a wash with a non-phosphate detergent, a rinse with tap water, and a final rinse with distilled water may be used. The equipment should be allowed to dry before use. Once decontaminated, the probes must be shown to be free of contaminants. At a minimum, a suitably sensitive organic vapor meter should be used for this purpose. Any probe that does not pass decontamination should not be used.
5. Handle and store decontaminated soil gas probes in a manner that prevents contamination.
6. Inspect each gas probe assembly for ware and faulty parts. Replace probe tips, O-rings, adapters, and probe rods as needed. New parts and parts in good working condition greatly reduce the chances of ambient air leaking into the soil gas sample and reduce the need for re-pushing probes.

Soil Gas Permanent Implant Installation

1. Assemble the drive point holder, implant anchor/drive point, and drive rod. Drive the rod to the desired bottom screen depth (for example, for a probe screened from 5 feet 6 inches to 5 feet bgs, the rod should be driven to 5 feet 6 inches). Do not disengage the drive point at this time.
2. Attach the 1/4-inch Teflon tubing to the probe screen. Use enough tubing so that at least 2 feet will be left above ground. Make sure that the tubing does not spin on the probe screen; if it does, it will not be possible to screw the probe screen into the drive point/anchor. Electrical tape can be used to secure the tubing to the screen. Plug the exposed end of the tubing with the probe cap.
3. Remove the drive head and thread the probe screen (Geoprobe Systems implant) and tubing down the inside of the drive rod. Once the implant reaches the drive point, turn the tubing counterclockwise with a gentle downward force to thread the screen into the drive point/anchor. Test that the screen is seated

by gently pulling up on the tubing. It is very important to ensure that the screen is seated before moving on to the next step.

4. Retract the drive rod 12 inches while pushing down on the Teflon tubing; this is to ensure that as the rod is being removed while the anchor/drive point and implant stay at depth.
5. Thread the tubing through a funnel and place the funnel on top of the drive rod. Determine the volume of glass beads needed to fill the space around the screen plus an additional 6-inch space above the screen. Remove the cap placed over the end of the tubing. Pour the beads into the funnel and down the inside diameter of the drive rod. Use the Teflon tubing to stir the glass beads and ensure they make it all the way down to the bottom. Do not pull on the tubing. Note: Failure to remove the cap during this step can result in bridging of glass beads (due to air displacement issue), and therefore an insufficient filter pack around screen. See Figure 1.
6. Lift the drive rod up an additional 18 to 24 inches and pour in the bentonite seal mixture. The mixture is 25 percent glass beads and 75 percent powdered bentonite clay (high-yield type). It takes approximately 154 milliliters of the mixture per foot. At least 2 feet of the mixture are recommended to adequately seal the hole and prevent contribution from ambient air during sampling. Pour 50 milliliters of water down the drive rod to initiate the bentonite seal.
7. Replace cap over end of tubing. Pull the drive rod the rest of the way out of the ground and fill the remaining hole to about 1 foot from the ground surface with either bentonite or cement.
8. Install either a stick-up or flush-mount cover to finish the probe. Coil the extra tubing inside the enclosure and cover. See Figure 2.
9. Wait at least 24 hours before sampling, so that the subsurface has time to equilibrate. Follow the proper procedures as presented in FOP 2, *Collection of Soil Gas Samples from Temporary and Permanent Probes Using Canisters*, and be sure that leak-check procedures are employed.
10. When calculating dead volume, use the internal volume of the Teflon tubing, the internal volume of the screen, and the volume of the glass bead pack (assume 30 percent porosity).
11. The surrounding ground surface shall be replaced and repaired to original condition.

FIGURE 1
Adding Glass Beads to the Geoprobe Implant

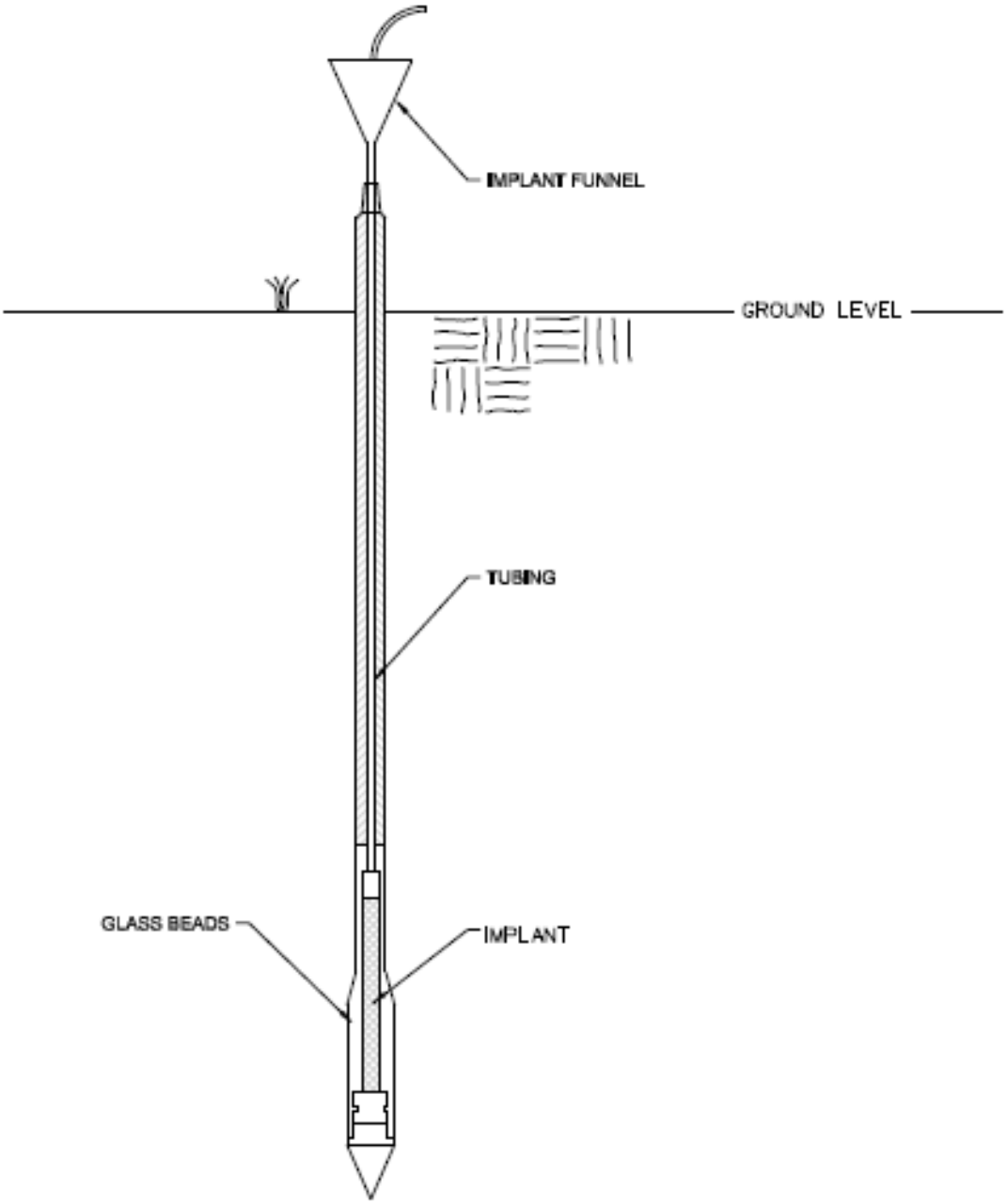
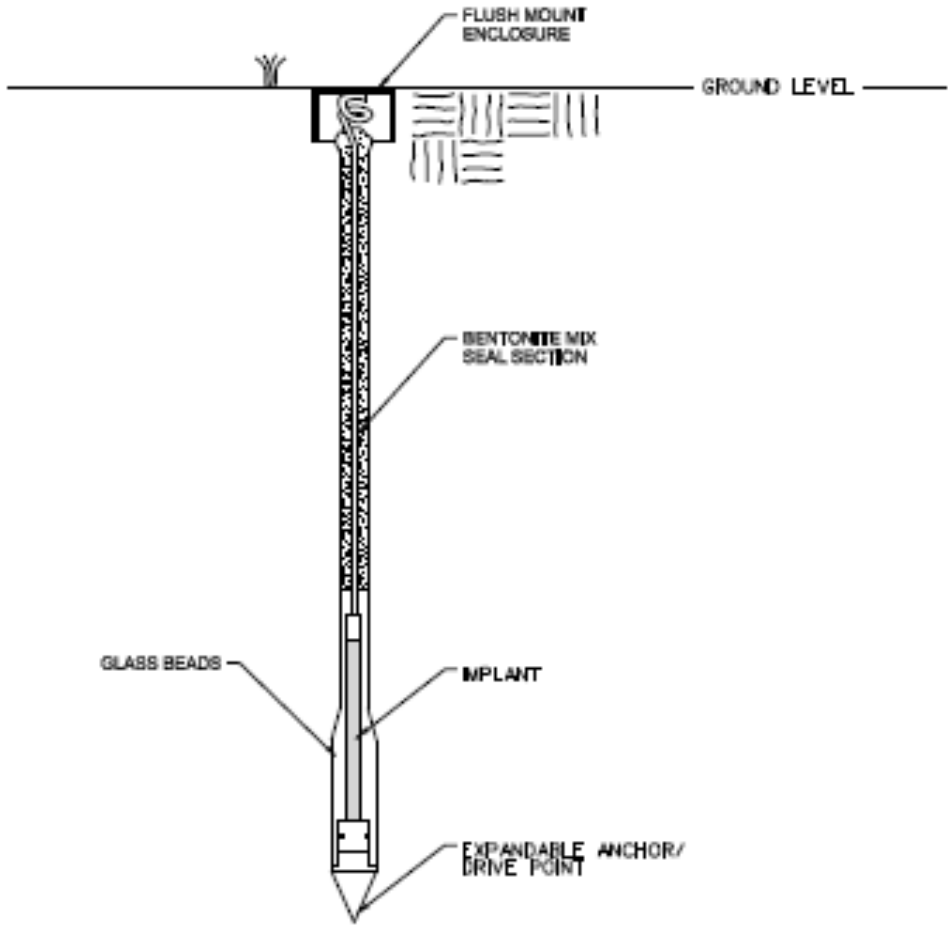


FIGURE 2
Installed Geoprobe Implant with Flush Mount Cover



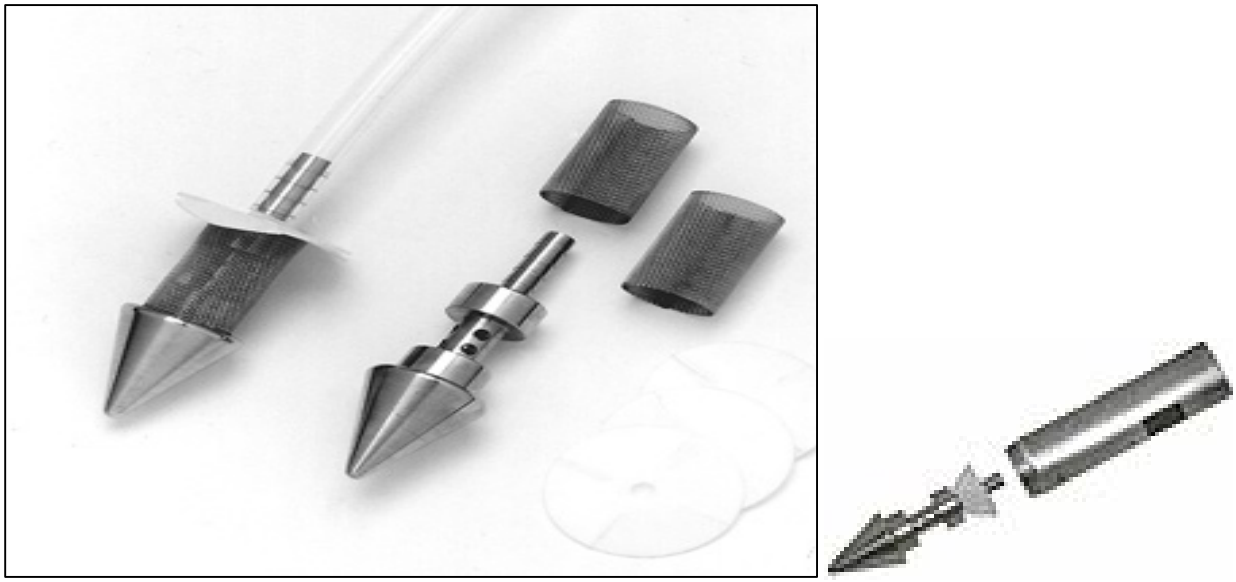
Soil Gas Probe Installation and Removal for the AMS Dedicated Gas Vapor Probe Method

1. Assemble the probe as shown in Figure 3 and attach the tubing. Thread the tubing through the hollow rod and attach the drive end to the top of the rod. Electrical tape may be used to secure the tubing to the tip, and protect the tubing from the drive end.
2. Push the probe to the desired depth with either the slide hammer or hammer drill. Attach extra rods to achieve the desired depth. Ensure that the final depth of the drive point includes extra depth to include the length of the retracted tip.
3. Retract the rod with the removal jack to expose the screen within the probe tip.
4. Install the probes in a manner that creates a leak-free seal between the aboveground atmosphere and the probe tip, while minimizing the impact on ground surface covers (such as asphalt, concrete, driveways, lawns) by following steps 5 to 7.
5. Determine the volume of glass beads needed to fill the space around the screen plus an additional 6-inch space above the screen. Remove the cap placed over the end of the tubing and thread the tubing through a funnel and place the funnel on top of the hole. Pour the glass beads into the funnel and down the inside of the hole. Use the Teflon tubing to stir the glass beads and ensure they make it all the way

down to the bottom. Do not pull on the tubing. Note: Failure to remove the cap during this step can result in bridging of glass beads (due to air displacement issue) and therefore an insufficient filter pack around screen.

6. Pour in a bentonite seal mixture an additional 18 to 24 inches on top of the glass bead filter pack. The mixture is 25 percent glass beads and 75 percent powdered bentonite clay (high-yield type). At least 2 feet of the mixture are recommended to adequately seal the hole and prevent contribution from ambient air during sampling. Pour enough water down the hole to initiate the bentonite seal.
7. Fill the remaining hole to about 1 foot from the ground surface with either bentonite or cement. Remove funnel and replace cap over end of tubing.
8. In the event the installation technique does not work, and a predrilled pilot hole is needed, this procedure must be coordinated with the project engineer. Use of predrilled holes will require careful control so as to not over-drill, and may also create the need for back-grouting to overcome leakage from the aboveground ambient atmosphere.
9. Wait 30 minutes after the probe is installed and sealed properly to begin sampling, so that the subsurface has time to equilibrate. The probe cap should be tightened on the end of the tubing during the equilibration period. Follow the proper procedures as presented in FOP 2, *Collection of Soil Gas Samples from Temporary and Permanent Probes Using Canisters*, and be sure that leak-check procedures are employed.

FIGURE 3
AMS gas vapor probe assembly



Attachment

1. Soil Boring Log

Attachment 1
Soil Boring Log

CH2MHILL	PROJECT NUMBER	BORING NUMBER	SHEET OF

SOIL BORING LOG

PROJECT : LOCATION : DATE:

WEATHER: DRILLING CONTRACTOR :

DRILLING METHOD AND EQUIPMENT USED :

WATER LEVELS START : END : LOGGER :

DEPTH BELOW SURFACE (FT)	INTERVAL (FT)	RECOVERY (IN)	#/TYPE	STANDARD PENETRATION TEST RESULTS 6"-6"-6"-6" (N)	CORE DESCRIPTION SOIL NAME, USCS GROUP SYMBOL, COLOR, MOISTURE CONTENT, RELATIVE DENSITY, OR CONSISTENCY, SOIL STRUCTURE, MINERALOGY.	COMMENTS DEPTH OF CASING, DRILLING RATE, DRILLING FLUID LOSS, TESTS, AND INSTRUMENTATION. OVM (ppm): Breathing Zone Above Hole

Sampler Signature: Date:

CH2MHILL	PROJECT NUMBER	BORING NUMBER	SHEET OF

SOIL BORING LOG

PROJECT : LOCATION : DATE:

NOTES

SOP(s) used (refer to SOP Log)?

Were all requirements of the SAP, PIs and above mentioned SOP(s) met?

Explanation of exceptions to SAP, PI's and SOP(s) *including why, under what conditions, who authorized exception, anything considered in the decision*:

Sampler Signature: _____ Date: _____

FIELD OPERATING PROCEDURE 2

Collection of Soil Gas Samples from Temporary and Permanent Probes Using Canisters

Purpose and Scope

This field operating procedure (FOP) offers a practical approach for the collection of soil gas samples from temporary soil gas probes (for instance, Geoprobe Systems direct-push system with post-run tubing [PRT] adapters or the AMS Retract-A-Tip system), or from permanently installed soil gas probes, into SUMMA (or equivalent) canisters. Soil gas sample integrity is verified by using a real-time leak-checking procedure before taking each sample. Verification must be done after probe installation and prior to sampling, as well as before each subsequent soil gas sample from permanent probes. This FOP should be used when its application is consistent with the project's data quality objectives (DQOs) and in conjunction with FOP 1, *Installation of Permanent Soil Gas Sampling Probes*. It is the responsibility of the project team to make sure this procedure meets all applicable regulatory standards and receives approval/concurrence from the leading regulatory agency for the project. Vapor intrusion (VI) subject-matter experts (SMEs) should be consulted as needed to address technical, regulatory, or field implementation issues associated with the use of this FOP. Only persons trained in the collection of soil gas samples should attempt this procedure.

Equipment and Materials

Typical equipment required includes the following:

- Teflon tubing 1/4-inch-outside-diameter (OD) sample tubing. Ensure there is enough tubing to use new tubing at each sample location.
- Swagelok 1/4-inch nut and ferrule sets for connecting the probe tubing to the sampling manifold (part #SS-400-NFSET).
- The helium leak-check equipment (or equivalent), including the enclosure, helium cylinder (high-purity helium [NO BALLOON HELIUM]), and helium detector (Dielectric MGD is preferred). The enclosure may be provided by the driller or can be constructed from polyvinyl chloride pipe. The helium detector can be rented from an equipment rental company.
- MultiRae five-gas meter (optional if onsite atmospheric gas analysis is required).
- Vacuum pump for purging with rotometer to control flow to 200 milliliters/minute (should be a Cole Parmer # R-79200-00 gray diaphragm pump or equivalent)
- Electric supply for the pump (either generator or power inverter with adapter for car battery).
- Sampling manifold consisting of Swagelok gas-tight fittings with three valves and one vacuum gauge to attach the probe to the air pump and the sample canister. See Figure 1. The manifold must be clean, free of oils, and flushed free of volatile organic compounds [VOCs] before use; this is accomplished by flushing three or four volumes of purge gas (ultra-high-purity air or nitrogen) through the manifold and associated tubing.
- Swagelok valve (only necessary for extended sampling periods [that is, more than 30 minutes]) so that the sampling manifold can be disconnected without introducing indoor air into the probe) (part # SS-4P4T)
- Wrenches (clean and free of contaminants), various sizes as needed for connecting fittings and making adjustments to the flow controller (if field-adjustable). A 9/16-inch wrench fits the 1/4-inch Swagelok fittings, which most canisters and flow controllers have.

- Gas sampling bag (such as Tedlar bag) (1-liter or 3-liter) to collect the purged soil gas, so the volume of purged soil gas can be measured and field screening with a photoionization detector (PID) or GEM2000 meter can be performed on the purged gas.
- GEM2000 landfill gas meter—optional if field measurements of CO_2, O_2, or CH_4 are necessary (aerobic biodegradation parameters typically measured for petroleum hydrocarbon sites).
- MiniRae PID meter for health and safety to ensure breathing zone VOC concentrations remain below levels specified by the health and safety plan. It is also optional to collect field measurements of total VOCs from the probe or purged soil gas; it may warn the laboratory if high concentrations are detected so they can dilute the sample before analysis.
- Canister—stainless steel, polished, certified-clean, and evacuated. Canisters are typically cleaned, evacuated, and provided by the laboratory.
- Flow controller or critical orifice, certified clean and set at desired sampling rate. These are typically cleaned, set, and provided by the laboratory. Soil gas samples are typically collected in 1- or 6-liter canisters at a flow rate of 200 milliliters per minute; however, lesser flow rates may be used in finer-grained soils.
- Negative pressure (vacuum) gauge—oil-free and clean—to check canister vacuum. The vacuum gauges are typically provided by the laboratory. The laboratory may either provide one vacuum gauge to be used with all of the canisters, or a vacuum gauge for each canister to be left on during sample collection. Sometimes the canisters are fitted with built-in vacuum gauges that are not removable. Gauges sent by the laboratory are for field use only, and are an approximate measure of the actual vacuum. Regularly calibrated—and less rugged—vacuum gauges are used at the laboratory to measure vacuum before shipment and again after sample receipt.
- A CH2M HILL-owned and -maintained PG5 Digital Pressure Gauge from Automation Products Group (APG) with National Institute of Standards and Technology (NIST)–traceable calibration certificate, or equivalent. The digital vacuum gauge will be used to check the initial and final negative pressures of the canisters.
- Shipping container, suitable for protection of canister(s) during shipping. Typically, strong cardboard boxes are used for canister shipment. The canisters should be shipped to the laboratory in the same shipping container(s) in which they were received.

Procedures/Guidelines

1. Soil gas sampling should not be performed until 48 hours after a significant rain event (defined as more than 1 inch of rainfall).
2. It is common practice to both install and remove soil gas probes by the direct-push method using equipment specifically designed for this purpose (Geoprobe or equivalent drill rig). Operation of direct-push machinery will be performed only by trained and licensed personnel. Soil gas probes can also be advanced with a hand tool method (such as the AMS Retract-A-Tip system). The hand tool installation method is only applicable to relatively shallow sampling (for example, up to 10 to 15 feet below ground surface [bgs], depending on the soil type).
3. Methane and carbon dioxide (CO_2) can cause positive bias with a helium leak detector, if a helium leak-check procedure is used as detailed in this FOP. If methane or CO_2 are expected or encountered at a site, then it may be necessary to explore different strategies to determine well integrity.
4. The subsurface needs time to equilibrate after probe installation; 30 minutes for temporary probes and 24 hours for permanent probes.
5. Prior to attempting sampling of soil gas probes, there should be an understanding of subsurface conditions at the site, as follows:

5.1. Depth to groundwater—Soil gas samples must be collected in the vadose zone (and above the capillary fringe).

5.2. Soil permeability—It may not be feasible to collect soil gas from finer-grained or tight soils with little pore volume, such as clays; if there are clay layers present in the subsurface, the intervals should be avoided. For sampling in soils, using permanent soil gas probes with a wider borehole is recommended. Care should be taken during purging and sampling so that the vacuum in the sampling system never exceeds 7 inches of mercury (inches Hg) or approximately 100 inches water.

System Setup

1. Acquire all the necessary hardware and sampling equipment shown in Figure 1. Be sure to place the helium leak-check enclosure over the probe, remove the probe cap, and push the sample tubing through the hole in the enclosure cap before attaching the sampling manifold. It may be necessary to cut off the probe cap with tubing cutters if the hole in the enclosure cap is not large enough to accommodate the ferrule set. The nut and probe cap can be reused once sampling is complete. The ferrule set must be replaced. Do not connect the canister at this time.
2. Adjust the purge system evacuation pump sampling rate to achieve the desired flow rate of 200 milliliters per minute or less. Flow rate measurements should be performed at the outlet of the vacuum pump prior to purging, either by using a suitable flow meter or by determining the amount of time required to fill a 1-liter gas sampling bag.
3. If the sample will be collected over a period of time more than 30 minutes a flow diversion valve (Swagelok part# SS-4P4T) should be placed in-line between the probe and the manifold. Once purging has been completed, the flow diversion valve can be turned to the off position, allowing disconnection of the manifold and vacuum pump for use at another location, without the loss of purge integrity at the purged location.
4. Sampling canisters are evacuated prior to shipment by the laboratory. The vacuum must be verified in the field with the APG Digital Pressure Gauge *and* the laboratory-supplied vacuum gauge, and must be properly recorded prior to use.
5. Attach the air pump to the sampling manifold and the Tedlar bag to the air pump exhaust. See Figure 2. Do not attach the canister at this time.

FIGURE 1
Sampling Manifold

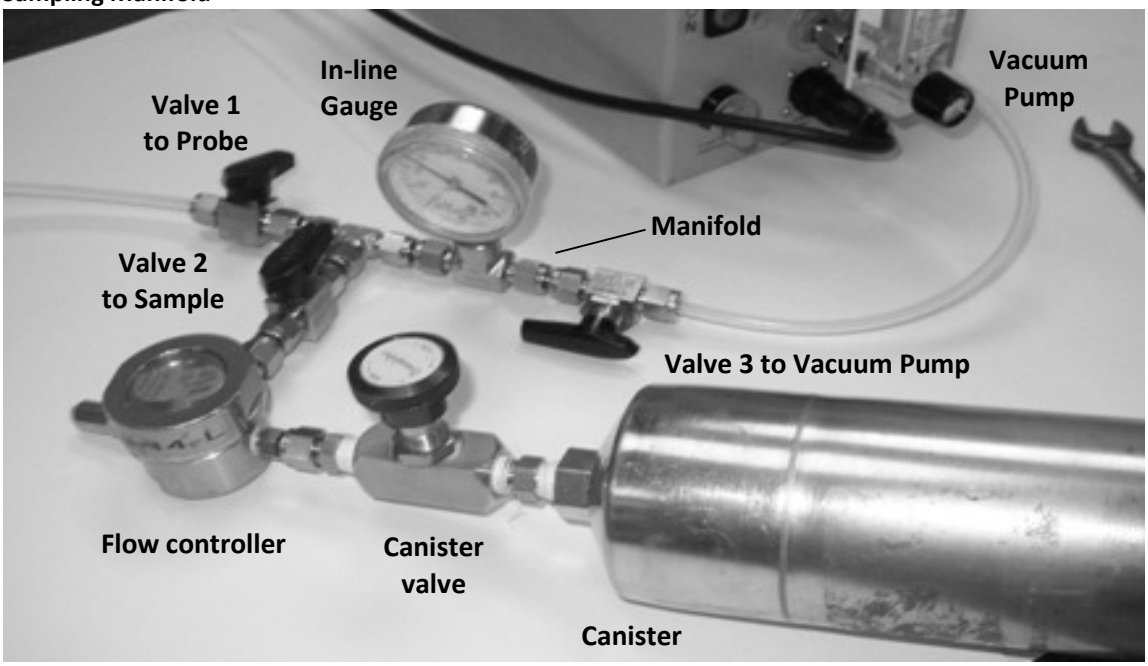
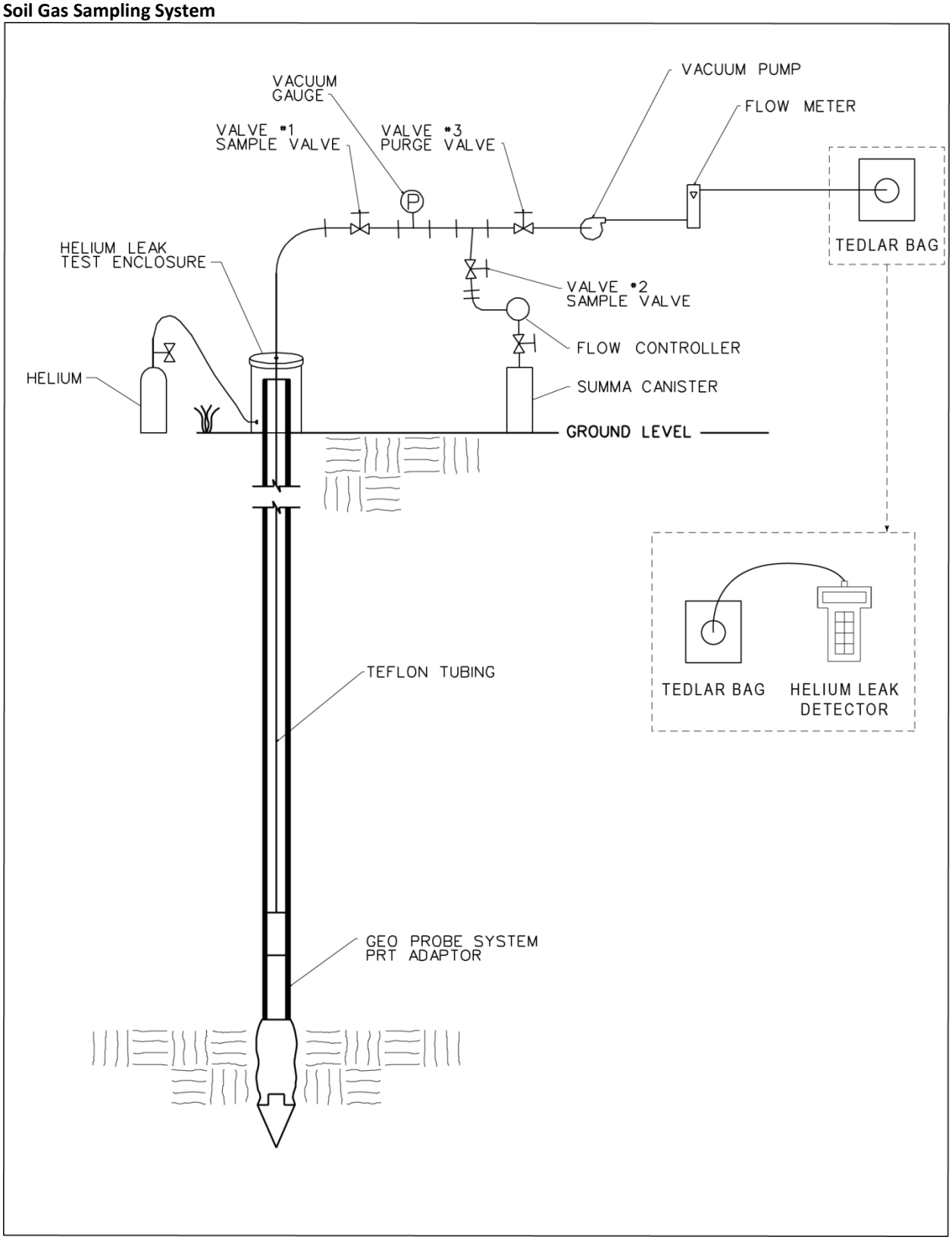


FIGURE 2
Soil Gas Sampling System



Soil Gas Sampling System Leak Checking and Purging Procedure

1. Physical Leak Check—Perform a leak check of the sample manifold system (Figure 1):

   1.1. Make sure the gas probe valve (valve #1) is closed and the sample valve (valve #2) is open.

   1.2. Open the purge valve (valve #3) and start the vacuum pump. Verify that the flow is set to 200 milliliters per minute.

   1.3. Close the sample valve (valve #2) and achieve a vacuum gauge reading of 10 inches Hg or to a vacuum that will be encountered during sampling, whichever is greater.

   1.4. A leak-free system will be evident by closing off the purge valve (valve #3), turning off the vacuum pump, and observing no loss of vacuum within the sampling manifold system for a period of 30 seconds. Repair any leaks prior to sample collection by tightening the fittings on the manifold. Re-test to make the sure the manifold passes the physical leak check before proceeding.

   1.5. Record the leak check date and time on the soil gas sampling log.

2. System Purge and Helium Leak Check—A purge of the soil gas probe and sampling manifold system is required before taking each sample. The helium leak-check procedure is also performed during this step. This helium leak check will verify the integrity of the sampling adapter (or PRT adapter if using the Geoprobe Systems system) seal as well as the probe and ground interface; this is accomplished by completing the following steps:

   2.1. Place the helium leak-check enclosure around the soil gas probe to achieve a buildup of helium in the leak-check enclosure. The enclosure should not be sealed tightly, and there should be an exhaust for the helium so pressure does not build up in the enclosure. Where the ground surface is soft, the helium leak-check enclosure is pressed down slightly into the ground surface. In situations where the ground surface is hard (for example, asphalt), apply a slight downward pressure to achieve a buildup of helium in the leak-check enclosure.

   2.2. Start the flow of helium under the leak-check enclosure at 200 milliliters per minute. Try to position the tube so the helium is directed at the interface between the probe and the ground. Let the helium fill the enclosure for a couple of minutes.

   2.3. Turn the helium leak detector on while in outdoor air and ensure that the detector is not reading any helium before proceeding. Verify that the helium concentration inside the leak-check enclosure is more than 10 percent by placing the probe of the helium detector into the hole where the sample tubing comes out or under the enclosure wall. It is not necessary to verify that the helium concentration is 100 percent, as this may damage the detector.

   2.4. Purging is carried out by pulling soil gas through the system at a rate of 200 milliliters per minute for a time period sufficient to achieve a purge volume that equals 3 to 5 dead volumes (internal volume) of the in-ground annular space, sample line, and sampling manifold system. When calculating the dead volume, be sure to take into account the inside diameter and length of the Teflon sample tubing, as well as the probe outside diameter and retraction distance for the annular space of temporary probes. For permanent probes, calculate the volume of the annular space using a nominal 30 percent porosity for the sand or glass bead pack. If, during the purge (or sampling), the vacuum exceeds 7 inches Hg, then reduce the pump flow rate. The system vacuum must stay below this level at all times to minimize the risk of inducing leaks or altering conditions in the subsurface.

   2.5. Open the sample valve (valve #2) and the purge valve (valve #3) and start the purge pump. Verify that the flow rate is still 200 milliliters per minute.

   2.6. To start the soil gas probe purge, simultaneously open the gas probe valve (valve #1) and close the sample valve (valve #2), and start timing.

2.7. If there is shallow groundwater in the area, carefully watch the tubing as the pump is turned on. If water is observed in the sample tubing, **shut the pump off immediately**. Soil gas collection will not be feasible if the probe is in contact with water.

2.8. Connect the helium detector to the enclosure exhaust to confirm that helium is present in the enclosure during purging. It is optional to measure the helium concentration within the enclosure (see Step 5.2.12). Make sure that the helium detector is exposed to ambient air and "zeros out" before measuring the purged soil gas in Step 2.12.

2.9. During the last 5 minutes of the purge (or the entire purge time if less than 5 minutes), attach a gas sampling bag to the purge pump exhaust and open the bag's valve. This bag/sample will be used for analysis of helium using the leak detector.

2.10. If the vacuum gauge reads more than 7 inches Hg during the purge, then close the purge valve (valve #3) and monitor the vacuum in the manifold and probe. If there is no noticeable change in vacuum after a minute, then there is an insufficient amount of soil gas to collect a sample and the vacuum is too great to collect a soil gas sample. Several factors can cause this situation, including the following (consult with the project manager and take corrective action):

2.10.1. The soil formation is too "tight" (that is, high clay or moisture content). Try using a lower flow rate (temporary or permanent probe).

2.10.2. The soil formation is too tight. Try a different depth or location (temporary probe).

2.10.3. With a temporary probe system, the expendable tip may not have released when the drive rod was retracted. Try retracting the probe a little further, or use a long, thin rod to poke the tip loose.

2.10.4. If water is visible in the flexible soil gas tubing, stop the purging immediately. It is not possible to take a soil gas sample at that depth or location.

2.11. At the end of the calculated purge time and after the system is verified to be leak free, close the purge valve (valve #3), close the valve to the gas sampling bag, and turn off the pump. Do not open the purge valve again. Doing so will result in loss of the purge integrity and will require re-purging.

2.12. Attach the gas sampling bag to the helium detector using a piece of flexible silicone or rubber tubing and open the valve. The helium concentration in the purged soil gas must be less than 1 percent of what it was in the helium enclosure during purging to pass the leak test (10,000 parts per million by volume [ppmv] if the helium concentration was 100 percent) (verify that the limit is consistent with appropriate project-specific regulatory guidance). Either (1) calculate what 1 percent of the helium concentration was in the enclosure from the measured concentration in Step 7.2.6, or (2) use a limit of 0.1 percent (1,000 ppmv), which allows for a 10-times safety margin. If the probe fails the leak check, then corrective action is required; this includes first checking the fittings and connections and trying another purge and leak check. It may also be necessary to remove the soil gas probe and re-install it in a nearby location.

2.13. Note: Helium leak detectors may be sensitive to high concentrations of methane or other atmospheric gasses. If high concentrations are expected to be present in the soil gas, then caution should be used with this technique, as false positive readings may be encountered during leak testing. Use a GEM2000 landfill gas meter to determine if methane is present in subslab soil gas.

2.14. At the end of the purge and after the system is verified to be leak-free, close the purge valve (valve #3). Do not open it again. Doing so will result in loss of the purge integrity and will require re-purging. Turn off the helium leak detector.

2.15. The purged subslab soil gas in the Tedlar bag can be screened with a GEM2000 landfill gas meter to get field measurements of CO_2, O_2, and CH_4 and/or a MiniRae PID can be used to measure concentrations of total VOCs in the field.

2.16. Record the purge and leak check information on the soil gas sampling log.

2.17. Immediately move on to the sampling phase. Little to no delay should occur between purging and sampling.

Sample Collection

1. Clean sampling protocols must be followed when handling and collecting samples. The protocols require care in the shipping, storage, and use of sampling equipment. Cleanliness of personnel who come in contact with the sampling equipment is also important—for example: no smoking, eating, or drinking; no perfumes or deodorants; and no dry-cleaned clothing. Canisters should not be transported in vehicles with gas-powered equipment or fuel cans. Permanent markers should not be used for labeling or note-taking during sampling.

2. The air sampling canisters are certified clean and evacuated by the laboratory to about 29 to 30 inches of mercury vacuum. Initial canister vacuums that are less than certified by the laboratory are a potential indication of leakage that could affect the accuracy of analytical results. Care should be used at all times to prevent inadvertent loss of canister vacuum. Never open the canister's valve unless the intent is to collect a sample or check the canister vacuum with an attached gauge.

3. Verify that the canister has sufficient initial vacuum for sampling. Measure the initial canister vacuum using an external vacuum gauge, as follows:

   3.1. Remove the protective cap from the valve on the canister. Make sure the canister knob is closed!

   3.2. Measure the initial canister pressure using a digital vacuum gauge with 0.25 percent accuracy at the -30 to 0 inches Hg range and NIST-traceable calibration for vacuum measurements. See the *Technical Bulletin: Use of External Vacuum Gauges with Canisters* for a recommended model of vacuum gauge[1] for use with Summa canisters used for VI sampling. Open the canister knob and record the reading. Close the canister knob and remove the digital vacuum gauge.

   3.3. If using assigned analog vacuum gauges (one for each canister), attach the vacuum gauge to the canister, and then attach the flow controller.

   3.4. Do not sample using a canister without sufficient initial vacuum. Be advised that sampling data may be flagged or rejected from canisters with low initial vacuum (less than 28 inches Hg). Low initial vacuum could create a low bias in analytical results due to air leakage. While there is also a smaller risk that air leakage could introduce contaminants into the canister, the primary concern is the low bias to analytical results; this bias is within the range of analytical variability allowed with U.S. Environmental Protection Agency (USEPA) Method TO-15 (±30 percent) for initial vacuums more than 24 inches Hg. The following table identifies the field team's response based on the initial vacuum reading for a canister. In addition, the table also identifies the potential bias to results at different initial canister vacuums.

[1] A PG5 Digital Pressure Gauge from Automation Products Group (APG), Inc. (http://www.apgsensors.com/products/pressure-sensors/digital-pressure-gauges/pg5) with National Institute of Standards and Technology (NIST)–traceable calibration certificate, or equivalent, is recommended for making vacuum measurements.

3.5. Use the following table to determine when to use canisters based on initial vacuum readings.

Initial Vacuum Reading	Potential Error in Analytical Results Due to Leakage	Field Team Response
More than 30 to 28 inches Hg	Up to -10% error	Use canister for sampling—no limitations on use.
More than 26 to 28 inches Hg	Up to -21% error	Use canister for sampling if necessary; replace canister with a spare if spares are available.
More than 24 to 26 inches Hg	Up to -30% error	Sampling with canister is not advisable. Contact project manager and obtain direction before sampling with this canister. Be advised that qualifiers may be applied to analytical results sampled with canisters with vacuums less than 26 inches Hg.
Less than 24 inches Hg	>-30% error	Do not use this canister for sampling. Analytical results will be rejected.

4. Attach the canister to the flow controller and then connect the flow controller to the sample valve (valve #2) on the sampling manifold. Open the sample valve (valve #2).
5. Before collecting the sample, confirm that the sampling system valves are set as follows: (1) the purge valve (valve #3) is confirmed to be closed; (2) the gas probe valve (valve #1) is open; and (3) the sample valve (valve #2) is open.
6. Slowly open (counter-clockwise) the canister's knob approximately one full turn.
7. Each sampling canister that is left unattended should be clearly marked with a sign that includes contact information for a point of contact. The sign can be edited with project-specific information, laminated, and attached to each sampling canister using cable ties (do not attach the signs using adhesive tape).
8. After sampling for the appropriate amount of time (determined from project instructions), close the sample valve (valve #2) and the canister's knob. If the canister has a built-in or assigned vacuum gauge, allow the canister to fill until the vacuum reaches 2 to 10 inches Hg for 6-liter canisters and 2 to 5 inches Hg for 1-liter canisters and record the final pressure. Remove the canister from the sampling manifold.
9. Measure the final canister pressure using the same digital vacuum gauge used to measure initial canister pressure. Open the canister knob and record the reading. Close the canister knob and remove the digital vacuum gauge.
10. If using a single laboratory provided external vacuum gauge, re-attach it, open the canister knob, and record the final vacuum. Close the valve, remove the gauge, and replace and tighten the cap on the canister. Ideal final vacuum in the canister is between 2 and 10 inches Hg. More than 10 inches Hg of vacuum can greatly increase reporting limits; however, a small amount of vacuum should be left in the canister so the laboratory can confirm that the canister was not opened during shipment.
11. Consult with the project manager and the project-specific VI senior technical consultant (STC) before submitting the sample to the laboratory if a final vacuum greater than 10 inches Hg, or less than 2 inches Hg are encountered.

Use the following table for guidance to determine how to address final vacuum measurements:

Final Vacuum Reading	Field Team Response
Less than 2 inches Hg	Contact the project manager and project VI STC before submitting sample. Notify the analytical laboratory to report the laboratory-measured pressure and to get direction from the project manager before analyzing sample.
More than 2 inches Hg and less than 10 inches Hg	Submit sample for analysis—no limitations on data use.
More than 10 inches Hg	Contact project manager and project VI STC before submitting sample. Verify final vacuum with the analytical laboratory before analysis.

12. Canisters with no vacuum left (that is, 0 inches Hg) should not be analyzed. Contact the project manager and project VI STC before submitting a sample with a final vacuum of 0 inches Hg to determine the appropriate course of action. One option is to verify the final vacuum with the analytical laboratory. If there is vacuum remaining in the canister according to the laboratory vacuum gauge, the project manager and/or project VI STC may direct the analytical laboratory to analyze the sample.
13. The analytical laboratory should be directed to not analyze a sample showing a final vacuum of 0 inches Hg (as measured by the laboratory), and to notify the project manager and obtain further guidance regarding that sample.
14. Record the sampling date, times, canister identification (ID), flow controller ID, vacuum gauge ID(s), and any other observations pertinent to the sampling event on the soil gas sampling log. Also record the weather conditions (temperature, barometric pressure, precipitation, etc.) during sampling.
15. Fill out all appropriate documentation (sampling forms, sample labels, chains of custody, sample tags, etc.).
16. Disassemble the sampling system.
17. For permanent probes, replace the probe cap and make sure it is securely in place. For temporary probes, remove the probe and abandon the bore hole.

Altitude Correction

1. Air pressure decreases with elevation. Therefore, a canister evacuated at a laboratory located at sea level will show a lower vacuum measurement at a higher altitude. Generally, a 1,000-foot rise in elevation corresponds to a 1-inch-Hg drop in pressure *or* a 1-inch-Hg decrease in measured vacuum. For example, a canister evacuated to 30 inches at sea level and used at 3,000 feet would show an initial vacuum of 27 inches Hg.
2. If you plan to sample at altitude, be sure to inform the laboratory ahead of time so it adjusts the flow controllers accordingly.
3. If sampling is being conducted at higher elevations, verify the elevation difference between the analytical laboratory and field location and determine the associated decrease in measured vacuum.
4. Calculate the pressure difference between the laboratory and field location as follows: Difference from Sea Level (field) minus Difference from Sea Level (laboratory). Use the Altitude Correction Table (Attachment 2).
5. Subtract the pressure difference determined in step 4 from allowable initial vacuum levels and final vacuum levels to determine appropriate initial and final vacuum levels.

Sample Handling and Shipping Procedure

1. Fill out all appropriate documentation (chain of custody, sample tags) and return canisters and equipment to the laboratory.
2. The canisters should be shipped back to the laboratory in the same shipping container in which they were received. The samples should not be cooled during shipment. DO NOT put ice in the shipping container.
3. When packing the canisters for shipment, verify that the valve (just past finger-tight) and valve caps are snug (one-quarter turn past finger-tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces. Never pack the canisters with other objects or materials that could cause them to be punctured or damaged. Ensure that flow controllers and gauges are separately and adequately wrapped to prevent damage.
4. Do not place sticky labels or tape on any surface of the canister.
5. Place a custody seal over the openings to the shipping container.
6. Make sure to insure the package for the value of the sample containers and flow controllers if corporate card policy does not cover the insurance.
7. Ship canisters for overnight delivery. NOTE: If sampling on a Friday, ensure the laboratory accepts samples on Saturdays (you do not want the canisters sitting on some loading dock [or worse] for 3 days).

Quality Control

1. Laboratories supplying canisters must follow the performance criteria and quality assurance prescribed in USEPA Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. Standard operating procedures are required.
2. Laboratories supplying flow controllers must follow the performance criteria and quality assurance prescribed in USEPA Method TO-14/15 for flow controller cleaning and adjustment. Standard operating procedures are required.

Attachments

1. Exterior Soil Gas Probe Installation and Sampling Log—Canister Method
2. Altitude Correction Table

Attachment 1
Exterior Soil Gas Probe Installation and Sampling Log—Canister Method

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Sheet ___ of ___

Vapor Intrusion Best Practices
Exterior Soil Gas Probe Installation and Sampling Log - Canister Method

Project Info

Project Name: ..... **Project # :** .....

Sampler Name: ..... **Date:** .....

Site

Identification: .....

Address: .....

Site Information:

Describe ground cover .....

Depth to groundwater (feet below ground surface) .....

Describe vadose zone soil type(s) .....

Was a soil boring log completed? ..... Was a probe diagram completed? .....

Soil Gas Probe Installation, Purging, Leak Checking, & Sampling Log

Sample location (describe and show in diagram)		
Probe and Sample Identification (field ID)		
Probe Installation	Date and time	
	Depth of hole drilled (feet below ground surface)	
	Bottom of probe screen (feet below ground surface)	
	Length of probe screen (inches)	
	Width of probe screen (inches)	
	Dead volume - including screen, sand pack, and tubing (mL)	
Manifold Leak Check	Leak check (sampling manifold) - Pass/No Pass	
Probe Purge	Purge rate (mL/min)	
	Purge start time	
	Purge vacuum (" Hg)	
	Purge completion time	
Helium Leak Check*	Leak check (% or ppmv helium)	

Field Analysis (optional)	GEM2000 - O2 (%)	
	GEM2000 - CO2 (%)	
	GEM2000 - CH4 (%)	
	PID - Total VOCs (ppmv)	
Canister Sampling	Canister ID	
	Flow controller ID	
	Pressure gauge ID (optional)	
	Sampling rate or period (mL/min or hours)	
	Sample start date and time	
	Initial canister pressure (" Hg)	
	Sampling vacuum (" Hg)	
	Sample completion date and time	
	Final canister pressure (" Hg)	

* The soil gas probe passes the helium leak check if the detected helium concentration is less than 1,000 ppm (0.1%). Do NOT collect a soil gas sample if the probe fails the helium leak test.

Weather conditions during sampling: .....

.....

.....

Observations and Comments: .....

.....

.....

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Vapor Intrusion Best Practices
Soil Gas Field Sampling Log

Sheet ___ of ___

Diagram - Outline of Site & Location of Soil Gas Probes

N

Note:
Show the location of each soil gas probe and indicate distances from significant features at the site.

Other observations and comments: .....

.....

.....

.....

.....

.....

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Attachment 2
Altitude Correction Table

ATTACHMENT 2

Altitude Correction Table

Elevation (feet)	Pressure (inches Hg)	Difference from Sea Level (inches Hg)	Elevation (feet)	Pressure (inches Hg)	Difference from Sea Level (inches Hg)
0	29.92	0	1500	28.37	1.553
50	29.87	0.053	1550	28.32	1.603
100	29.81	0.106	1600	28.27	1.653
150	29.76	0.159	1650	28.22	1.703
200	29.71	0.212	1700	28.17	1.753
250	29.66	0.265	1750	28.12	1.803
300	29.60	0.317	1800	28.07	1.853
350	29.55	0.370	1850	28.02	1.903
400	29.50	0.422	1900	27.97	1.953
450	29.45	0.474	1950	27.92	2.002
500	29.39	0.527	2000	27.87	2.052
550	29.34	0.579	2050	27.82	2.101
600	29.29	0.631	2100	27.77	2.151
650	29.24	0.683	2150	27.72	2.200
700	29.19	0.735	2200	27.67	2.249
750	29.13	0.787	2250	27.62	2.298
800	29.08	0.838	2300	27.57	2.347
850	29.03	0.890	2350	27.52	2.396
900	28.98	0.941	2400	27.47	2.445
950	28.93	0.993	2450	27.43	2.494
1000	28.88	1.044	2500	27.38	2.543
1050	28.82	1.095	2550	27.33	2.591
1100	28.77	1.147	2600	27.28	2.640
1150	28.72	1.198	2650	27.23	2.688
1200	28.67	1.249	2700	27.18	2.736
1250	28.62	1.299	2750	27.14	2.785
1300	28.57	1.350	2800	27.09	2.833
1350	28.52	1.401	2850	27.04	2.881
1400	28.47	1.452	2900	26.99	2.929
1450	28.42	1.502	2950	26.94	2.977
			3000	26.90	3.025

Note: Use the following equation to calculate atmospheric for altitudes not shown on this table:

$P = P_o \exp(-35.523 \times 10^{-6}\, y)$, where P is the pressure at the desired elevation, P_o is the atmospheric pressure at sea level, and y is the desired elevation.

Source: National Aeronautics and Space Administration (NASA). 1996. *Elevation Correction Factor for Absolute Pressure Measurements*. NASA Technical Memorandum 107240.

FIELD OPERATING PROCEDURE 3

Surface Water Sampling

Purpose and Scope

This field operating procedure (FOP) presents the techniques used in collecting surface water samples.

Equipment and Materials

Materials and equipment vary depending on type of sampling. Typical equipment required includes the following:

- Open tube sampler
- Dip sampler
- Weighted bottle sampler
- Hand pump
- Kemmerer or Van Dorn sampler
- Depth-integrating sampler
- Peristaltic pump
- Sample containers
- Meters for specific conductance, temperature, pH, and dissolved oxygen

Procedures/Guidelines

Before surface water samples are taken, all sampler assemblies and sample containers will be cleaned and decontaminated as described in FOP 8, *Equipment Decontamination Procedures*. Methods for surface water sample collection are described in the following subsection.

Manual Sampling

Surface water samples are collected manually by submerging a clean glass, stainless steel, or Teflon container into the water body. Samples may be collected at depth with a covered bottle that can be removed with a tripline. The most common sampler types are beakers, sealable bottles and jars, pond samplers, peristaltic pumps, and weighted bottle samplers. Pond samplers have a fixed or telescoping pole attached to the sample container. Weighted bottle samplers are lowered below water surface, where the attached bottle is opened, allowed to fill, and pulled out of the water. When retrieved, the bottle is tightly capped and removed from the sampler assembly.

A sample is taken with the following specific steps:

1. The location and desired depth for water sampling are selected.
2. The sample site is approached from downstream in a manner that avoids disturbance of bottom sediments as much as possible. The sample bottle is gently submerged with the mouth pointed upstream and the bottle tilted slightly downstream. Bubbles and floating materials should be prevented from entering the bottle. If using a peristaltic pump, lower the tubing into the water to the desired depth.
3. For weighted bottle samplers, the assembly is slowly lowered to the desired depth. The bottle stopper is unseated with a sharp tug and the bottle is allowed to fill until bubbles stop rising to the surface.
4. When the bottle is full, it is gently removed from the water. If sample transfer is required, it should be performed at this time.
5. Measure dissolved oxygen, specific conductance, temperature, and pH at the sampling location.

Attachments

None.

Key Checks and Items

- Start downstream, work upstream.
- Log exact locations using permanent features.
- Beware of hidden hazards.

FIELD OPERATING PROCEDURE 4

Note-taking and Field Logbook

Purpose

The purpose of this field operating procedure (FOP) is to delineate protocols for recording field and sampling information in a field logbook.

Scope

Data generated from the use of this FOP may be used to support the following activities: site characterization, risk assessment, and evaluation of remedial alternatives.

Equipment and Materials

- Field logbook
- Indelible black ink pen
- Write-in-the-rain pen (for extreme weather conditions—cold/rain)

Procedures and Guidelines

All information pertinent to a field or sampling effort will be recorded in a bound field logbook that will be initiated at the start of the first onsite activity. The field logbook will consist of a bound notebook with consecutively numbered pages that cannot be removed. The outside front cover of the logbook will contain the project (site) name and the specific activity (for example, supplemental remedial investigation). The inside front cover will include the following:

- Site name and USEPA work assignment number
- Project number
- Site manager's name and mailing address
- Sequential logbook number
- Start date and end date of logbook

Each page will be consecutively numbered, dated, and initialed. All entries will be made in indelible black ink, and all corrections will consist of line-out deletions that are initialed and dated. If only part of a page is used, the remainder of the page should have an "X" drawn across it. At a minimum, entries in the logbook will include the following:

- Time of arrival and departure of site personnel, site visitors, and equipment
- Instrument calibration information, including make, model, and serial number of the equipment calibrated
- Description of significant activities for the day
- Documentation of photographs taken during field activities (for example, date, time, and description of photograph)
- Field observations (for example, sample description, weather, unusual site conditions or observations, sources of potential contamination, etc.)
- Detailed description of the sampling location, including a sketch when necessary
- Details of the sample site (for example, coordinates [*x*, *y*], water elevation [*z*], casing diameter and depth, integrity of the casing, etc.)

- Sampling methodology and matrix, including distinction between grab and composite samples
- Names of field team members and subcontractors
- Start or completion time of sample collection activities
- Field measurements (for example, water depths and sediment probe depths)
- Type of sample (for example, sediment, groundwater, surface water, soil and debris)
- Number, depth, and volume of sample collected
- Field sample number
- Requested analytical determinations
- Sample preservation
- Quality control samples associated with the sample
- Sample shipment information including chain-of-custody form number and laboratory, carrier, date, and time
- Health and safety issues (including level of personal protective equipment)
- Signature and date by personnel responsible for observations

Sampling situations vary widely. No general rules can specify the extent of information that must be entered in a logbook. However, records should contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The field team leader will keep a master list of all field logbooks assigned to the sampling crew.

Key Checks and Items

None.

FIELD OPERATING PROCEDURE 5

Utility Clearance for Intrusive Operations

Purpose

This field operating procedure (FOP) describes the utility mark-out/clearance process to be followed prior to completing any intrusive subsurface activities.

Scope and Applicability

This document describes the utility mark-out/clearance process to be followed by CH2M HILL prior to any intrusive activities. The document discusses steps that must occur prior to mobilizing and steps that must occur during intrusive work.

This document should be reviewed by the field project team prior to working in the field. In addition, the document should be used to develop scopes of work for utility clearance subcontractors.

Procedures/Guidelines

Prior to Mobilizing to the Field

Prior to mobilizing to perform intrusive work, the state one-call center, must be called and utility companies must mark out the utility lines.

Public Utilities

The state one-call center **must** be called prior to any intrusive work. The following general information must be provided during the call:

- Your name, company, address, and phone number where you can be reached, and the company doing the digging.
- The name and phone number of the site contact.
- The county and city or county and unincorporated area of the township of the excavation.
- The location of the intrusive work, which may include, but not be limited to, address, cross street, lot numbers, etc. At least one of the following must be provided: (1) appropriate section and quarter section grid information; (2) sufficient address or descriptive information to allow the establishment or drawing of a dig site polygon; (3) sufficient address, street, and cross-street information to allow for the determination of the appropriate section and quarter section grid(s); or (4) global positioning system (GPS) coordinates.
- The start date and time the planned activities.
- If subcontractors are responsible for the utility locate proper documentation must be received from them prior to the start of intrusive activities and placed in the project folder.

While on the phone with one-call, the following information should be collected and documented in the project files:

1. **Members Notified.** The identity of one-call members notified will be provided to the caller. Retain a copy in the project files and keep it onsite while intrusive work is being performed.
2. **Case Reference Number.** An identification number associated with the call should be retained for future reference, if needed. Retain a copy in the project files and keep it onsite while intrusive work is being performed.

The site safety coordinator should retain a copy in the project records and keep it onsite while intrusive work is being performed.

All public utilities in the area where intrusive work is to be performed should be marked out on the ground by the utility locator using the American Public Works Association Uniform Color Code (Attachment 1).

Utility locates are good for 21 calendar days, including the day the call was made. Extended tickets are available and will be extended, and one-call must be notified if extended tickets are needed.

Private Utility Clearance

Private utilities must be cleared as follows:

1. The location(s) where intrusive work will occur should be identified during a site visit with the designated utility locator. The proposed areas where intrusive work will be performed should be premarked by CH2M HILL before this site visit. It is important to take access issues into consideration while premarking.
2. The utility locator should clear an area for intrusive work. Additional area (if possible) to be cleared by the utility locator is based on the work to be performed.
3. The area that is cleared by the utility locator for intrusive work should include the marking of all utilities in the immediate area (at least a 10-foot buffer around the cleared area) using orange spray paint. If it is not possible to mark utilities, then a figure should be provided that will show the field team exactly where the utilities are located.
4. Record the name and telephone number of the representative conducting the utility clearances.
5. The utility clearance is applicable for a 30-day period. Any intrusive work conducted after this 30-day period requires a new utility clearance.

Prior to Intrusive Work

The following should be completed before commencing intrusive work:

1. Verify that all public utility companies have identified the presence of utilities with marking paint or have provided a response back indicating the absence of utilities in the area. To verify what the utility markings on the ground indicate, use the color code in Attachment 1 (for public utilities). If utilities have not been marked or a negative response has not been confirmed, do not perform intrusive work in that area. Contact one-call and alert them of the situation.
2. Review the utility clearance documentation with the subcontractor during the tailgate meeting.
3. Use other methods to identify utilities if there are numerous utility lines around the area and/or lines that cannot be clearly located where intrusive work is to be performed. If possible, hand-digging or hand-augering will be performed down to 5 feet below ground surface (bgs). Another method would involve the use of an air knife to bore 5 feet bgs with the use of high pressure air that would not damage any utilities encountered.
4. Intrusive work can only be performed in the cleared area. If intrusive work needs to be performed outside of the cleared area, the appropriate utility locator(s) must clear the new location. If the new area cleared involves private utilities, an addendum to the initial utility clearance signoff sheet should be provided.
5. While performing intrusive work, monitor for signs of an encounter with a utility line. These signs include encountering fill material such as gravel, sand, or other fill material; warning tape; plastic; or metal. If it is believed that a utility was struck stop work, call the appropriate personnel, and document in the field book.
6. If refusal occurs while boring and it is believed not to be related to a utility, then advancement will be tried up to two more times within cleared area (only applies to locations where the boring and a 3-foot radius was cleared). If the same refusal is observed, then the location will be abandoned.

Attachment 1

APWA UNIFORM COLOR CODE

FOR MARKING
UNDERGROUND UTILITY LINES

	PROPOSED EXCAVATION
	TEMPORARY SURVEY MARKINGS
	ELECTRIC POWER LINES, CABLES, CONDUIT AND LIGHTING CABLES
	GAS, OIL, STEAM, PETROLEUM OR GASEOUS MATERIALS
	COMMUNICATION, ALARM OR SIGNAL LINES, CABLES OR CONDUIT
	POTABLE WATER
	RECLAIMED WATER, IRRIGATION AND SLURRY LINES
	SEWERS AND DRAIN LINES

FIELD OPERATING PROCEDURE 6

Packing and Shipping of Environmental Samples

Purpose

The purpose of this field operating procedure (FOP) is to delineate protocols for the packing and shipping of samples to the laboratory for analysis.

Scope

This FOP is applicable for all samples collected and prepared for analysis at an offsite laboratory.

Equipment and Materials

- Waterproof hard plastic coolers
- Resealable plastic bags
- Plastic garbage bags
- Absorbent packing material (not vermiculite)
- Inert cushioning material (not vermiculite)
- Ice
- USEPA Region 5 sample tags
- Scribe software
- Laptop and printer
- Adhesive labels 2 by 4 inches (generated by Scribe software)
- Chain-of-custody forms (generated by Scribe software)
- USEPA Region 5 custody seals
- Air bills and shipping pouches (or example, Federal Express)
- Clear tape
- Strapping tape
- Mailing labels

Procedures and Guidelines

Prepare Bottles or Bags for Shipment

1. Arrange sample containers in groups by sample number.
2. Check that sample container lids are tight.
3. Secure appropriate USEPA Region 5 sample tags around lids of container with string or wire.
4. Arrange containers in front of assigned coolers.
5. Affix appropriate adhesive labels to each container.
6. Enclose each sample in a clear; resealable plastic bag, making sure that sample labels are visible.

Prepare Coolers for Shipment

1. Tape drains shut, inside and out.
2. Affix “This Side UP” labels on all four sides and “Fragile” labels on at least two sides of each cooler.
3. Place mailing label with laboratory address on top of the coolers.
4. Place inert cushioning material (for example, bubble wrap, preformed poly-foam liner) in the bottom of the cooler. Do not use vermiculite.
5. Place appropriate chain-of-custody records with corresponding custody seals on top of each cooler.

6. Double-bag and seal loose ice in resealable, plastic, zip top bags to prevent melting ice from leaking and soaking the packing material. Place the ice outside the garbage bags containing the samples. Place sufficient ice in cooler to maintain the internal temperature at 4±2 degrees Celsius during transport.
7. Put an absorbent pad in the bottom of the cooler and fill the cooler with enough packing material to prevent breakage of the sample bottles and to absorb the entire volume of the liquid being shipped (offsite sample shipment only).
8. Record the USEPA Region 5 custody seals on the chain-of-custody forms. Sign each chain-of-custody form (or obtain signature) and indicate the time and date the cooler was custody sealed.
9. Seal the laboratory copies of the chain-of-custody forms in a large resealable plastic bag and tape to the inside lid of the cooler. Retain the Region 5 copies of the chain-of-custody forms for return to USEPA. Each cooler must contain a chain-of-custody form (or forms) that corresponds to the contents of the cooler.
10. Close lid and latch.
11. Peel custody seals carefully from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
12. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals.
13. Relinquish to carrier (for example, Federal Express). Place air bill receipt inside the mailing envelope and send to sample documentation coordinator, along with the other documentation.

Key Checks and Items

None.

FIELD OPERATING PROCEDURE 7

Sample Handling and Chain-of-Custody Procedure

Purpose

The purpose of this field operating procedure (FOP) is to provide a definition of "custody" and describe protocols for documenting the transfer of custody from one party to the next (for example, from the site to the laboratory). A documented custody trail is established through the use of sample tags and a USEPA chain-of-custody form that uniquely identifies each sample container, and who has possession of it from the sample's origin to its final destination. The chain-of-custody form also describes the sampling point, date, time, and analysis parameters.

Scope

Sample personnel should be aware that a sample is considered to be in a person's custody if the sample meets the following conditions:

- It is in a person's actual possession.
- It is in view after being in a person's possession.
- It is locked up so that no one can tamper with it after it has been in physical custody.

When samples leave the custody of the sampler, the cooler must be custody-sealed and possession must be documented.

Data generated from the use of this FOP may be used to support the following activities: site characterization, risk assessment, and evaluation of remedial alternatives.

Equipment and Materials

- Computer with Scribe software loaded
- Laser printer with paper (8.5- × 11-inch) and ink cartridge (black)
- USEPA Region 5 Sample Tag
- Scribe generated tag label (2- × 4-inch adhesive labels)
- Indelible black ink pen

Procedures and Guidelines

Chain-of-Custody Forms

The chain-of-custody form (see Attachment 1 for example) must contain the following information:

- Case Number/Client Number: If a Contract Laboratory Program (CLP) laboratory is used, enter the case number provided by USEPA's Regional Sample Control Center Coordinator (RSCC). If the CLP is not used, enter the SAS number provided by CH2M HILL's sample and analytical coordinator.
- USEPA Region: Enter Region "5".
- Site Name/State:
- Project Leader: Enter the CH2M HILL site manager's name.
- Action: "Remedial Design"
- Sampling Co.: "CH2M HILL"

- Sample No.: This is the unique number that will be used for sample tracking. For CLP, this number is taken from a block of numbers assigned by the USEPA RSCC. For non-CLP, the CH2M HILL sample and analytical coordinator will assign the number.
- Matrix: Describes the sample media (for example, “Sediment”).
- Sampler Name: The name of the sampler or sample team leader.
- Concentration: Low (L), Low/Medium (M) or High (H).
- Sample type: “Grab” or “Composite”
- Analysis: This indicates the analyses required for each sample.
- Tag No.: This number appears on the bottom of the sample tag and includes a prefix (“5”) followed by a series of numbers. The entire number must appear on the chain-of-custody form.
- Preservative: Document what preservative has been added to the sample (for example, “HCl,” “Ice Only,” “None”).
- Station Location: This is the CH2M HILL Station Location Identifier.
- Sample Collect Date/Time: Use military time.
- QC Type: This is for field quality control (QC) only, and includes field duplicates.
- Date shipped: The date that samples are relinquished to the shipping carrier.
- Carrier Name: (for example, “FedEx”)
- Airbill: Air bill number used for shipping.
- Shipped to: This is the laboratory name and full address, including the laboratory contact. If the contact is not known, use “Sample Custodian”.
- Chain-of-Custody Record fields: The sampler’s signature must appear in the “Relinquished By” field. The date and time (military time) must also be included.
- Although the samples are “relinquished” to the shipping carrier, the shipping carrier does not have access to the samples as long as the shipping cooler is custody sealed. Consequently, the shipping carrier does not sign the chain-of-custody form.
- Sample(s) to be used for laboratory QC: This identifies which samples are to be used for matrix spike/matrix spike duplicate analyses.
- Indicate if shipment for case is complete: USE “Y” OR “N”.
- Chain-of-Custody Seal Number: Record the custody seal numbers that appear on the Region 5 custody seals that can be found on the shipping container. There is usually a minimum of two per shipping container.

Sample Tags

Each sample container will be identified with a uniquely numbered sample tag (see Attachment 1 for example) issued by USEPA Region 5. Each tag will contain the following information:

- Case/SAS number
- The unique sample number for sample tracking
- CH2M HILL station location (that is, the sample identifier)
- Date of sampling
- Time the sample was collected (in military time)

- All parameters for which the sample will be analyzed
- Preservative used (if any)
- Sample type (grab or composite)
- Sample concentration (low, medium, high)
- Sample matrix (soil, etc.)
- The signature of sample team leader
- Identification when sample is intended to be used by the laboratory for matrix spike (MS)/matrix spike duplicate (MSD)

Key Checks and Items

1. All sample containers must be properly tagged.
2. Each cooler must have a chain-of-custody form and the samples in the cooler (as identified by the sample tags) must match what is on the chain-of-custody form.
3. Each chain-of-custody form must be properly relinquished (signature, date, time).
4. The custody seal numbers must be written on each chain-of-custody form.
5. The shipping cooler must be custody sealed in at least two places.
6. Chain-of-custody forms will be completed with required sampling information, and recorded information will match the sample tags.
7. If the designated sampler relinquishes samples to other sampling or field crew members for packing or other purposes, the sampler will complete the chain-of-custody form prior to this transfer.
8. Appropriate personnel will sign and date chain-of-custody forms to document the sample custody transfer.
9. Original chain-of-custody forms will accompany the shipment; copies will be retained by the sampler for sampling records.
10. If samples are sent by common carrier, bills of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation.
11. Commercial carriers will not be required to sign off on chain-of-custody forms as long as the forms are sealed inside the sample cooler and the custody seals remain intact.
12. Packaging, marking, labeling, and shipping of samples will comply with the regulations promulgated by the U.S. Department of Transportation in the *Code of Federal Regulations* (49 CFR 171-177).

Attachment

Attachment 1: Scribe Quick Reference Guide

ATTACHMENT 1

Quick Guide to using Scribe

The following is a step-by-step guide for preparing and shipping samples to the CLP laboratory using Scribe.

Setting up Event

1) Open the Scribe Program.
   a) The program normally opens to the last project you were working.
   b) If the program does not open to the correct project file, go to:
      i) File
      ii) Open Project
      iii) Select “XXX.MDB”
2) Select “EVENTS” on the left bar under “Planning”
3) Select “ADD” from the top bar.
4) Fill in the event information and click “SAVE” at the bottom.
5) Go to “ANALYSES” on the left bar under “Planning”
   a) Scroll down to the analyses you need and input the TAT. Note that you need to input both the number and the units! (ex: 35 in the first column and days in the second column).
6) Your lab may not be saved into the system yet. Go to “LAB LIST” on the left-hand bar. If you do not see your lab, click “add” and put in the lab information.

Inputting Samples

1) Go to “Soil/Sediment” on the left hand bar under “Sampling”
2) Choose “Add” on the bottom bar of the screen.
3) Fill in your sample information (See **Figure 1**).

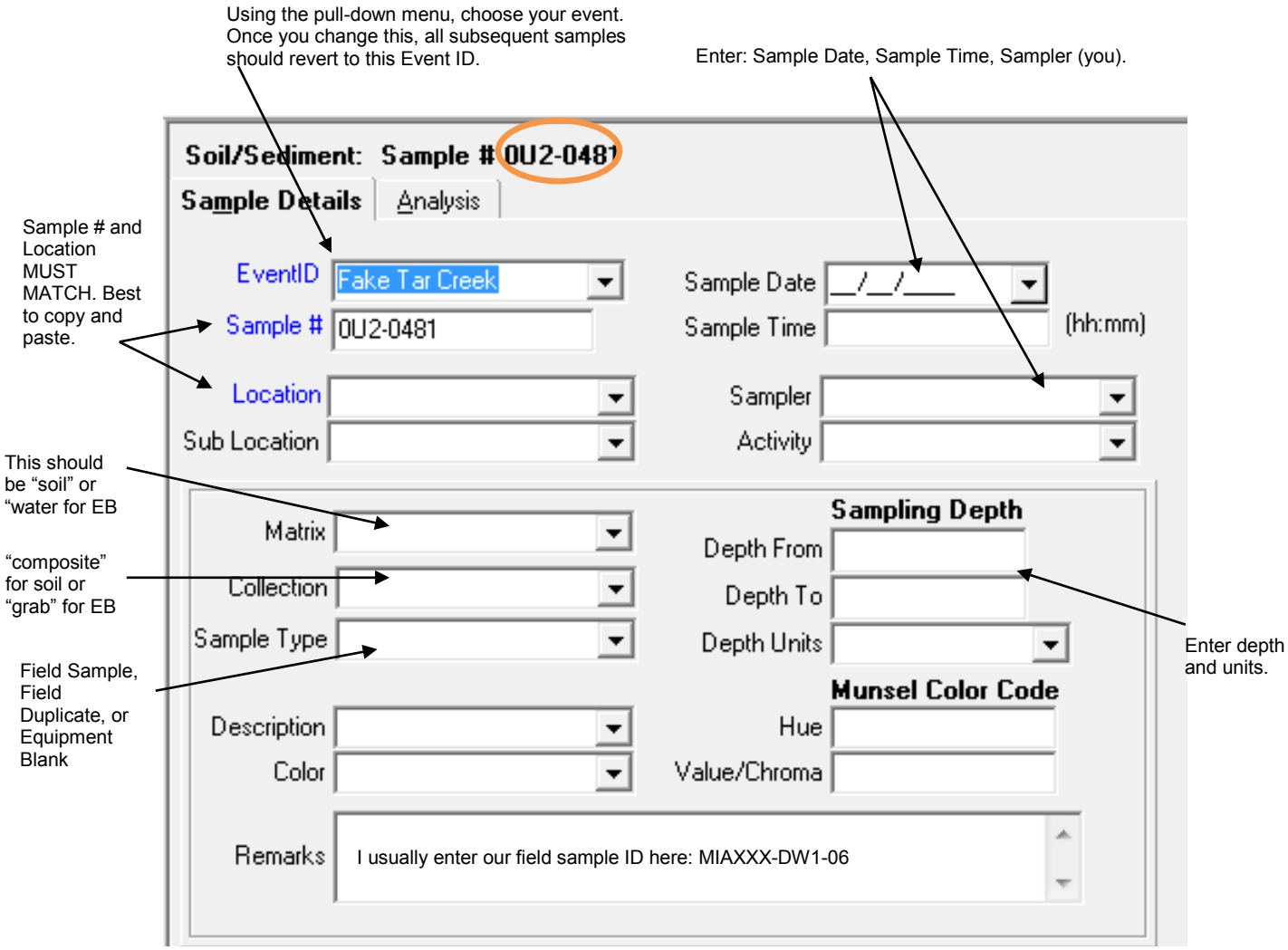

Figure 1. Adding a sample to the database.

4) Once you are done entering your information, click the "Analysis" tab (orange circle in **Figure 1**).
5) For your FIRST sample, you need to update your tag settings. After you do this, if all of your tags are in sequential order, you will not need to change this again.
   a) Click on "CLP/Tag" settings on the bottom bar of the screen.
      i) Update information to include the next tag number and make sure that the following boxes are checked:
         (1) Assign numeric tag numbers.
         (2) Use region number to prefix tag numbers of the COC.
      ii) Update the Case # for CLP.

   See **Figure 2**.

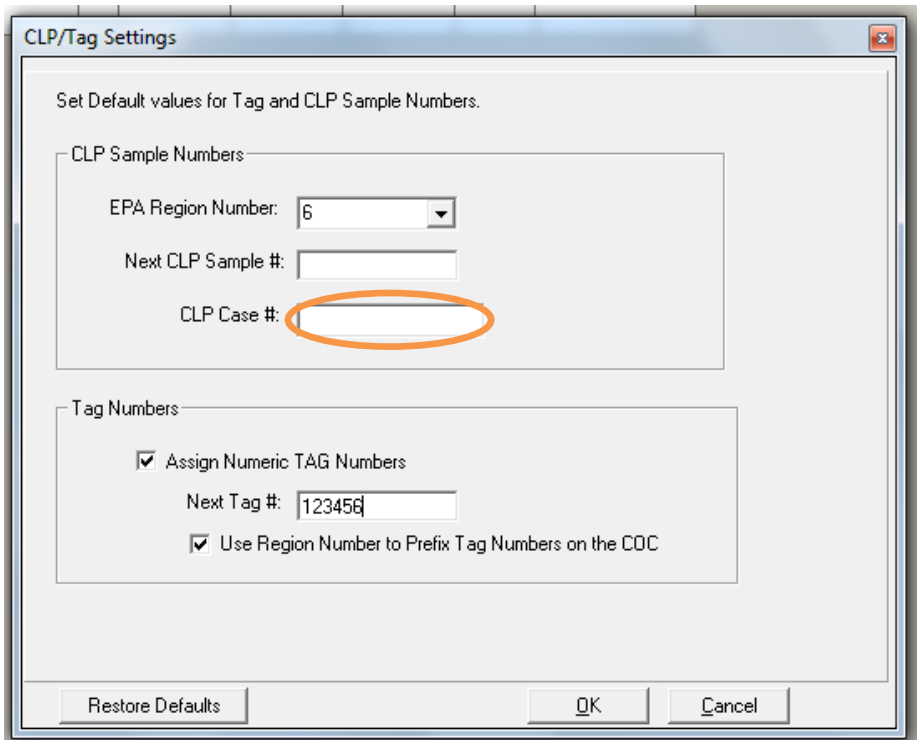

Figure 2. Tag settings

6) Now it's time to enter your analysis (**Figure 3**). Note: Even though we have two containers for each sample ID, you still only want to state that you have 1 sample because each sample has its own tag number. You need to fill out all field below (tag number will automatically update) except for Description. **FILL IN THE CLP SAMPLE # AND MAKE SURE IT IS ALSO PRESENT ON YOUR LABELS.**
7) When you have finished filling out the information, highlight the row and click "COPY ANALYSES" on the bottom bar of the screen. This will allow you to have 2 tag numbers for 2 jars under the same sample ID.

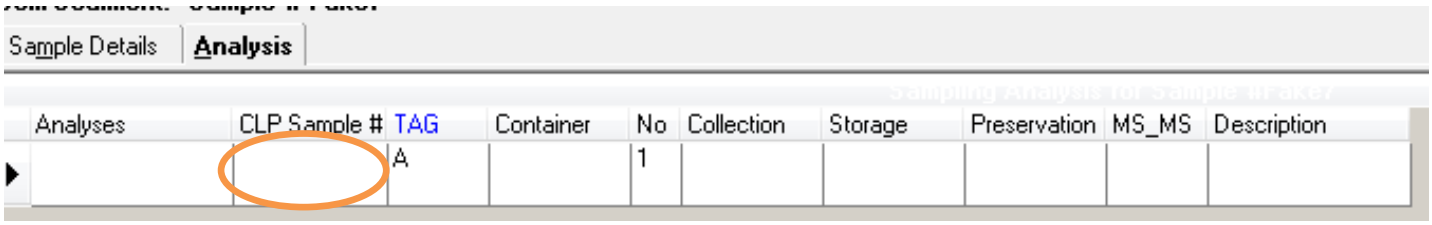

Figure 3. Filling out your analysis details.

8) Repeat steps 1–7 in the "Inputting Samples" section of this quick guide until all your samples have been entered. It is okay if you plan on shipping in different coolers, you do not have to decide anything yet. You are simply setting up your sample database.

Helpful Hints:

- Having trouble finding something in the "summary" view? Try changing which columns you can view. Go to "view" and then "select columns". This makes your QC a bit easier.
- Your most recent addition will be listed on the top row. Want to change your samples in sequential order by sample number? Use the sort function on the top bar and chose how you want to sort your samples.

- Have too many samples listed on the screen that aren't part of your event? Use the filter function on the top bar of the screen. Best bet is to filter for your Event ID.

Printing Labels

1) From the left-hand bar, choose "SAMPLES" under "SAMPLE MANAGEMENT"
2) Filter for your Event ID.
3) This is a great time to QC your work.
4) Once you have finished your QC, you are ready to print labels!
5) Sort your samples by tag # (this will make it easier to apply your stickers).
6) Highlight the rows you wish to print.
7) Click "PRINT LABELS" on the bottom of your screen.
8) Choose "LABEL SETUP"
9) System is already set up for our 5160 label
10) Select "NEXT" and you will see the label setup. Your label should include:
    a) **CLP Sample #**
    b) Tag #
    c) Location
    d) Preservative
    e) Analysis
    f) Date:
    g) Time:
11) Choose which label number you wish to start with.
12) "FINISH"
13) You can print this to stick-on labels or to standard paper and cut-and-paste to tags.
14) Print two sets of stickers (one for tags and one for jars).

The Chain of Custody

1) Make sure you have printed your air bill, you will need the information for the COC.
2) From the left-hand bar, choose "CHAIN OF CUSTODY" under "SAMPLE MANAGEMENT"
3) Choose "ADD A CHAIN OF CUSTODY" from the bottom bar on the screen.
4) Fill out all the information (**Figure 4**).

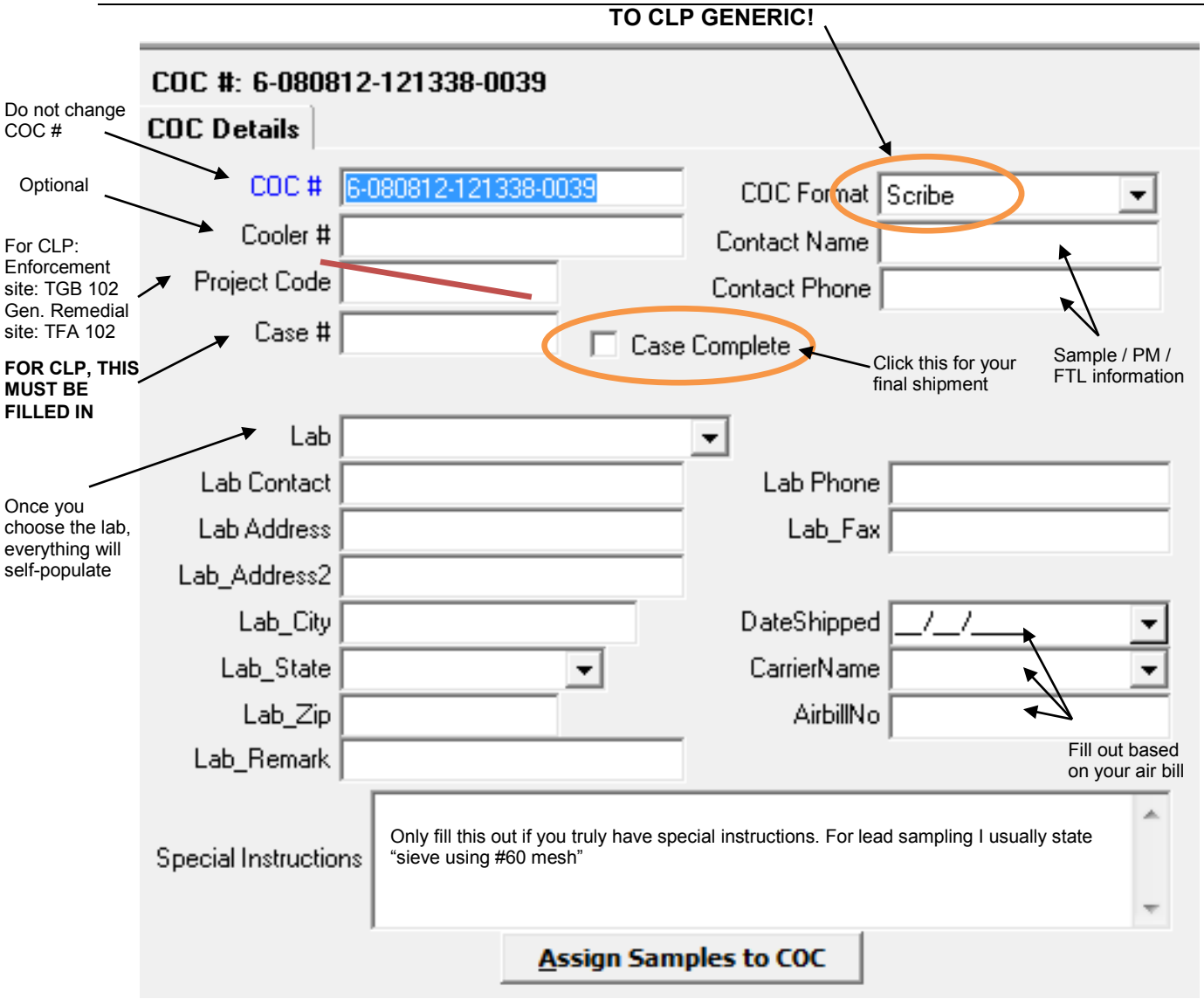

Figure 4. Filling out the COC information.

5) Click "ASSIGN SAMPLES TO COC"
6) You are now brought back to your samples screen
7) Filter for your Event ID
8) If you are shipping more than one cooler with the samples listed, filter further by filter for your sample ID. Double check that the correct samples are listed.
9) Highlight the samples you wish to add to the COC.
10) Click "ASSIGN to COC #" on the bottom bar of the screen.

Final Paperwork

1) Print your COCs. From the same screen you were on at the previous step, you should see "PRINT CHAIN OF CUSTODY" at the bottom of the screen. If you do not, go to "CHAIN OF CUSTODY" under "SAMPLE MANAGEMENT" and double click on the row with your COC#.
   a) Go to report setup and print the lab copy.
   b) Repeat the process for the region copy *(Note: the Region copy does not go in the cooler. At the end of the sampling event it is sent to Dave Shekoski/MKE for submittal to EPA.)*
2) Exporting your .XML File
   a) From the top bar on the screen choose "EXPORT"
   b) Choose "COC XML File" (the bottom option). This then gets uploaded to the "SMO WEB PORTAL"

FIELD OPERATING PROCEDURE 8

Equipment Decontamination Procedures

Purpose

The purpose of this field operating procedure (FOP) is provide general guidelines for the decontamination of personnel, sampling equipment, and monitoring equipment used in potentially contaminated environments.

Scope

This FOP is a general description of decontamination procedures.

Equipment and Materials

Distilled or deionized water

2.5 percent (W/W) Alconox, Liquinox, or equivalent phosphate-free detergent and water solution

Large plastic pails or tubs for Alconox, Liquinox, or equivalent and water, scrub brushes, squirt bottles for detergent solution, resealable plastic bags, paper towels, and sheets

55-gallon drum for disposal of waste, approved by the Department of Transportation (DOT)

Unpowdered chemical-resistant gloves (that is, nitrile gloves)

Aluminum foil

Procedures and Guidelines

Personnel Decontamination

The following procedures are to be performed after the completion of tasks whenever the potential for contamination exists, and also upon leaving the exclusion zone:

Wash boots in detergent solution, and then rinse with water. If disposable latex booties are worn over boots in the work area, remove and discard into a DOT-approved 55-gallon drum.
Remove and discard outer chemical-resistant gloves into a DOT-approved 55-gallon drum.
Remove disposable coveralls (“Tyveks”) and discard into a DOT-approved 55-gallon drum (if worn).
Remove respirator (if worn). Dispose of filter cartridges and replace daily.
Remove inner gloves and discard.
Shower entire body at the end of the work day, including hair, either at the work site or at home.
Sanitize respirator if worn.

Sampling Equipment Decontamination—Other Equipment

Reusable sampling equipment is decontaminated after each use as follows:

1. Wear unpowdered chemical-resistant gloves.

Rinse and scrub with potable water.
Wash all equipment surfaces that came into contact with the potentially contaminated soil/water with detergent solution.
Rinse with potable water.
Rinse with distilled water.
Completely air dry or wipe dry with a clean paper towel. Wrap exposed areas with aluminum foil (shiny side out) or enclose equipment in clean plastic for transport and handling if equipment will not be used immediately.
Collect all rinsate and place in a DOT-approved 55-gallon drum.

Dispose of decontamination materials (for example, plastic sheeting, tubing, etc.) that have come in contact with used decontamination fluids or sampling equipment in DOT-approved 55-gallon drums.

Health and Safety Monitoring Equipment Decontamination

1. Wrap soil contact points in plastic before use, to reduce need for subsequent cleaning.
2. Wipe all surfaces that had possible contact with contaminated materials with a paper towel wet with detergent solution, and finally two times with a towel wet with distilled water. Dispose of all used paper towels in a DOT-approved 55-gallon drum.

Sample Container Decontamination

The outside of sample bottles or containers filled in the field may need to be decontaminated before being packed for shipment or handled by personnel without hand protection. The procedure for sample container decontamination is as follows:

1. Wipe container with a paper towel dampened with detergent solution, or immerse in the solution AFTER THE CONTAINERS HAVE BEEN SEALED. Repeat the above steps using potable water.

Dispose of all used detergent solution and paper towels in a DOT-approved 55-gallon drum or in trash bags placed into appropriate dumpsters, keeping liquids and solids in separate drums.

Key Checks and Items

1. Do not use acetone for decontamination.

Drum all contaminated rinsate.

Clean with solutions of Alconox, Liquinox, or equivalent phosphate-free detergent, and distilled water.

Appendix B
Analytical Standard Operating Procedures

Title: Determination of VOCs in Ambient Air EPA Compendium Methods TO14 and TO15

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1.0 Scope and Application

This SOP describes the laboratory procedure for the analysis of polar and non-polar volatile organic compounds (VOCs) in ambient air. The procedure is applicable to those VOCs that have been evaluated by the laboratory for their consistent performance in meeting the control criteria put forth in Compendium Method TO-15. While the compendium method is specifically written for the analysis of samples collected in leak-free passivated stainless steel canisters, it may be applied to the analysis of samples that have employed the use of other collection devices such as Tedlar bags and solid absorbents.

1.1 Analytes, Matrix(s), and Reporting Limits

The target compound list and reporting limits for each compound are provided in Table 1.

2.0 Summary of Method

An aliquot of sample is pulled from the canister through a solid multi sorbent bed trap which reduces the water content of the sample. The sample is thermally desorbed and the VOCs are carried onto a GC column coupled to a mass spectrometer. Compounds are identified by comparison of the mass spectra for individual peaks in the total ion chromatogram to the fragmentation patterns of ions corresponding to VOCs including the intensity of primary and secondary ions as well as the patterns of stored spectra acquired under similar conditions. The concentration of the target compound is calculated by internal standard technique using the average response factor of that compound as determined by the initial calibration.

This procedure is based on EPA Compendium Method TO-15 "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.

If the laboratory has modified the method, a list of these modifications may be found in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Contamination may occur if canisters or other equipment is not properly cleaned before use. The laboratory procedures for canister and flow controller cleaning procedures are provided in Appendices C and D.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples

and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The analytical system contains zones with elevated or depressed temperatures that are capable of causing injury upon direct contact. The analyst needs to be aware of the locations of those zones, and allow them to return to room temperature prior to maintenance activities or take measures to avoid contact with hot and/or cold surfaces. There are areas of high voltage in the analytical system. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Liquid nitrogen (LN2) is used for cryogenic purposes. In addition to avoiding contact with LN2 cooled surfaces, analysts must be aware of the potential for oxygen depletion in a confined space in the event of an unexpected large release of the product. Users should evacuate a confined space in which large amounts of LN2 have been released.

Sample canisters are occasionally pressurized for cleaning or sample dilution purposes. Lab systems are designed to ensure that the cans are not pressurized above 40 psi. Eye protection must be worn when cans are pressurized in the event of a canister failure.

5.2 Primary Materials Used

There are no materials used in this method which have a serious or significant hazard rating **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment meets the specification of this SOP.

6.1 Sampling Equipment

- 6L, 3L, and 1L SUMMA® Canisters: Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 Valves, or equivalent.

- 6L SUMMA® Canisters: Silicon lined-Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 valves or equivalent.

- Flow Controllers: Restek Catalog #24239 or equivalent.

- Flow Controller Orifice: Various sizes ranging from 0.008” to 0.060”, Restek or equivalent.

- Flow Controller Vacuum Gauges: Capable of measuring vacuum to an absolute vacuum of -30” of HG, and pressure up to 30 psi, Grainger Catalog #5WZ37 or equivalent.

- Rain Guard: Stainless Steel Tubing ¼", 10ft. Grainger or Equivalent. Cut 8" and bend into a J shape using a pipe bender.

- Stainless Steel Pre-Filter (7 um): Swagelok Catalog# SS-4F-T7-7 or equivalent.

- Teflon Tape: Home Depot Brand or equivalent.

6.2 Analytical System

- Mass Spectrometer: Agilent 5973 or 5972 MSD or equivalent.
- Gas Chromatograph: Agilent 6890 or equivalent.
- VOC Autosampler: Entech 7016CA or equivalent.
- Cryogenic Concentrator: Equipped with an electronic mass flow controller that maintains a constant flow for carrier gas and sample over a range of 0-200 cc/min. Entech 7100A or equivalent.
- Low Pressure Liquid Nitrogen: Air Gas or equivalent.
- Glass Bead Cryotrap: Capable of effectively removing water while trapping polar and non-polar compounds. Entech catalog# 01-04-11320.
- TENAX Sorbent Trap: Capable of removing CO2 and trapping the polar and non-polar compounds. Entech catalog # 01-04-11330.Primary Column: Fused silica capillary column (60 m x 0.32 mm x 1.8 µm), Restek RTX-624 or equivalent.
- Data System: PC software for Entech instrumentation. Hewlett-Packard ChemStation data acquisition software and Hewlett-Packard ChemServer, Target 3.5 data processing software or TestAmerica Chrom and TestAmerica LIMS (TALS).

6.3 Cleaning System

- Canister Cleaner Module and Software: Capable of filling canisters with humidified air and evacuating canisters to 50 mtorr, Entech Model 3100A or equivalent.

- Vacuum Pump: Capable of evacuating sample canisters to full vacuum. Vacuubrand or equivalent.

- Cleaning Manifold: Equipped with stainless steel and Teflon transfer lines and connections for cleaning up to twelve canisters simultaneously.

- Heating Belts: Individual thermal-stated heating belts used to heat canisters to 100ºC during the manifolds cleaning cycles. Entech or equivalent.

- Cleaning oven: Capable of cleaning 6 Summa Cans simultaneously at a temperature of 100ºC. Entech or equivalent.

- Flow Controller Cleaning Manifold: Capable of flushing hot Nitrogen through 24 flow controllers simultaneously for cleaning.

6.4 Miscellaneous Supplies

- Mass Flow Controller, NIST Traceable: Capable of flow rate of 70 mL/min, McMillan Company 80SD or equivalent. Use for the preparation of calibration and working standards.

- Zero Air Generator: Ballston Model HPZA-3000 or equivalent.
- Syringes: Gas tight with a Luer-Lok tip, assorted sizes ranging from 1.0 mL to 1.0 L, SGE or equivalent.

- Digital Pressure Gauges, NIST Traceable: Capable of measuring pressure in the range of -30" Hg to 100 psi, Dwyer Models DPGA-12 and 67100 or equivalent.

- Digital Flow Meter, NIST Traceable: Alltech or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Ultra Pure Humidified Zero Air - Pass ambient laboratory air through a zero air generator. The zero air generator humidifies the air to a relative humidity of >20 percent.

7.2 Standards

Purchase the following stock standard mixtures from commercial vendors:

- Mixed Gas Stock Standard: - Commercially prepared standard that includes internal standard and tune standard compounds: Bromofluorobenzene, Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5, and at a concentration of 100 ppbv each. Spectra Gas or equivalent.

- Calibration Stock Standard: - Commercially prepared custom gaseous stock standard used by all network facilities that includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases or equivalent.

- Calibration Ethanol Neat Material. >99.5 %

- ICV / LCS Stock Standard: - Custom made gaseous stock standard prepared from a different lot(s) of the source material(s) used to manufacture the calibration stock standard. The ICV/LCS stock standard includes all target analytes at a concentration of 1.0 ppmv. Spectra Gases.

- ICV/LCS Ethanol Neat material. >99.5% from a source other than the calibration source.

Prepare calibration and working standards mixtures by diluting a known volume of the stock standard in humidified ultra pure zero air to a specified volume. The formulations for standard preparation are provided in Appendix B along with recommended expiration dates and storage conditions.

Each stock standard is assigned a 1 year expiration date from manufacture and recertified annually. The ethanol neat material is assigned the expiration date given by the manufacturer.

The recertification procedure is as follows:

Internal Standard Mixed Gas Stock: Assay the active internal standard cylinders against a new, vendor certified cylinder purchased from Spectra Gas. Verify that the % difference between new, vendor certified cylinder and the active cylinder is within 20%. If this criterion is not met, replace the cylinder.

Calibration Stock Standard and the ICV/LCS Stock Standard: Return one of the cylinders to Spectra for recertification (rotate a different cylinder each year). When the re-certified stock standard cylinder is returned, assay against all active cylinders. The difference between the recertified value and the assay values should be within 15%. If these criteria are not met, return the cylinder that was not re-certified to the vendor for recertification or purchase a new stock standard.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Air	1L ,3L or 6L Passivated Summa Canister	1L	NA	30 days from collection	EPA TO-15

All samples should be collected in passivated stainless steel canisters that have been certified clean prior to sampling. The laboratory will provide certified clean canisters to the client upon request. The procedures for clean canister certification are provided in Laboratory SOP BR-AT-011.

The laboratory can also provide flow controllers set to the appropriate flow rate for the sampling time required by the client.

The laboratory ships air canisters in custom made boxes. The boxes are equipped with custom-made foam inserts to hold the pre-set flow-controllers. The custom shipping materials are designed to prevent damage of equipment to and from the sampling site. The laboratory checks the equipment to ensure it is in proper working order before shipment to the client and additional checks are performed on return of the equipment to the laboratory. Sampling instructions are provided with each sampling kit. The sampling crew is advised to handle the sampling equipment using the instructions provided by the laboratory to ensure optimum performance.

Samples should be stored at ambient temperature.

The analytical holding time is 30 days from the date of sample collection.

9.0 Quality Control

9.1 Sample QC

The following quality control samples are prepared with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See Table 3
Internal Standard (ISTD)	Every Sample	See Table 3
Laboratory Control Sample Duplicate (LCSD)	Client request	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3
Trip Blank (TB)	Client Request	See Table 3

NOTE: The compendium reference method does not require the analysis of a laboratory control sample (LCS) or provide criteria for the evaluation of an LCS. The laboratory performs an LCS at the above mentioned frequency as an evaluation of percent recovery in a blank matrix. The control limits set by the laboratory for the LCS (70-130) are those specified in Section 11.4 of the reference method for the audit accuracy evaluation.

The compendium method does not require analysis of a LCSD. Evaluations for precision should be derived from field samples. Duplicate precision should be measured by the analysis of a sample duplicate. Replicate precision should be measured from separate aliquots taken from the same sample canister. The laboratory will perform an LCSD to measure precision only per client request and analysis of the LCSD is considered a billable. The acceptance criteria for duplicate and replicate precision is <25%. Unless otherwise specified by the client during project initiation, the LCSD will be used to measure precision only. The LCS will be used for evaluations for percent recovery and to determine if corrective action is necessary.

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Tune Standard (BFB)	Each Analytical Window	See Section 10.0
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Section 10.0
Initial Calibration Verification (ICV)	Once, after each ICAL	See Section 10.0
RT Window Establishment	Once per ICAL	See Section 10.0
Relative Retention Time (RRT)	With each sample	See Section 10.0
Continuing Calibration Verification (CCV)	Daily, after each BFB	See Section 10.0

10.0 <u>Procedure</u>

10.1 Support Equipment Calibration

Verify the calibration of the mass flow controller used to prepare standards, the calibration of the digital flow meter used to set and check the flow rates of the FC(s) used for sample collection, and the calibration of the digital pressure gauges used to check return canister pressure is

current to the year. Immediately notify the QA department if the calibration is not current and wait for further instruction. Equipment whose calibration has expired may not be used without documented approval from the QA department.

NOTE: The QA department schedules the annual calibrations of the support equipment and maintains all Certificates of Calibration. The flow controllers are checked against a NIST traceable standard. This check is performed by the manufacturer of the equipment, when possible, or by an approved vendor that provides certification service.

10.2 Instrument Calibration

10.2.1 Tune Standard

Analyze a tune standard (BFB) prior at the beginning of each analytical window. The tune standard is a commercially prepared mixed gas stock standard that includes bromofluorobenzene (BFB) at a concentration of 100 ppbv.

To analyze the tune standard:

1) Establish the instrument operating conditions specified in Section 10.4.1.

2) Attach the mixed gas stock standard to the Entech concentrator by attaching the cylinder to the line dedicated for introduction of the internal standard (ISTD). The concentrator directly injects 20 mL of the 100 ppbv stock standard onto the instrument to yield an on column concentration of 10 ppbv.

3) Acquire the data and evaluate the results against the acceptance criteria given in Table 2. Criteria must be met prior to further analysis. The official start time of the 24 hour analytical window is the injection time of a passing tune standard. All samples must be injected within 24 hours of that time.

NOTE: The data processing system averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of BFB.

10.2.2 Initial Calibration (ICAL)

The instrument must be calibrated with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method.

The laboratory routinely analyzes 8 standards at the recommended concentrations of 0.04, 0.20, 0.50, 5.0, 10.0, 15.0, 20 and 40 ppbv, except for Ethanol. For Ethanol, a 6 point curve is analyzed at the following concentrations: 5, 10, 15, 20, 40, and 100 ppbv. Even though seven calibration standards are routinely analyzed not every calibration standard is used for each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five standards. The analyte group associations for each target analyte are provided in Table 1. The calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven point calibration curve. The calibration range is 0.20 to 40 ppbv with the 0.04 ppbv standard routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.20 ppbv

- Group B: This analyte group is associated with a six point calibration curve. The calibration range is 0.50 to 40 ppbv with the 0.04 and 0.20ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 0.50 ppbv.

- Group C: This analyte group is associated with a five point calibration curve. The calibration range is 5.0 to 40 ppbv with the 0.20, 0.50, and 0.04 ppbv standards routinely excluded. The limit of quantitation (LOQ) for this group of analytes is 5.0 ppbv.

- Group D: This analyte group is an eight point calibration curve. The calibration range is 0.04 40 ppbv. The limit of quantitation (LOQ) for this group of analytes is 0.04 ppbv.

- Group E: (Ethanol : This analyte has a six point calibration curve. The calibration range is 5 to 100 ppbv. The limit of quantitation (LOQ) for this analyte is 5 ppbv.

Prepare the calibration standards using the formulations provided in Appendix B.

Analyze the standards in a sequence from lowest to highest concentration using the instructions provided in Section 10.4.2.

The data processing system calculates a relative response factor (RRF), for each analyte and isomer pair using the assigned internal standard. The internal standard associations for each target analyte are provided in Table 1. The data processing system also calculates a mean relative response factor, relative standard deviation (RSD), relative retention time (RRT) and the mean RRT.

The following criteria must be met for a calibration to be considered acceptable:

- The RSD for each target analyte must be <30% with at most 2 exceptions up to a limit of 40%**.**

- The area response for the primary quantitation ion for the internal standard for each ICAL standard must be within 40% of the mean area response over the calibration range for each internal standard.

- The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

If these criteria are not met inspect the system for problems and perform corrective action. Recommended corrective actions are provided in Section 10.2.5 and in Table 3.

Repeat initial calibration whenever instrument operating conditions are changed, a new column is installed, when significant instrument maintenance has been performed, and when the result of the CCV indicate the calibration is no longer valid.

10.2.3 Second Source Calibration Verification (ICV)

Immediately following an acceptable initial calibration verify the accuracy of the calibration by the analysis of the second source calibration verification standard (ICV).

Prepare the ICV following the formulation provided in Appendix B.

Analyze the ICV following the instructions provided in Section 10.4.2.

The percent recovery (%R) for each target analyte must be within 70-130%. If criteria are not met, perform corrective action. Recommended corrective actions are provided in Table 3. If corrective action is not successful, remake your standards and recalibrate.

If after successful analysis of the ICV, time remains in the 24-hour analytical window, QC and field samples may be analyzed without analysis of a continuing calibration verification check standard. If time does not remain in the analytical window, a new analytical sequence must be initiated with a Tune Standard followed by daily calibration (CCV).

10.2.4 Continuing Calibration Verification (CCV)

Analyze the CCV immediately after the tune standard unless the analytical window includes ICAL, in which case, a CCV is not required.

Prepare the CCV standard using the formulation given in Appendix B. The recommended concentration of the CCV for each target analyte is 10.0 ppbv.

Analyze the CCV following the instructions provided in Section 10.4.2. The data system calculates a response factor for each analyte and calculates the percent difference (%D) of the RRF relative to the mean RRF in the most recent initial calibration.

- The %D for each target analyte must be within ±30%. If the above criteria are not met, repeat the analysis of the CCV <u>once</u>. If the second CCV meets criteria, continue with the analytical sequence. If it fails, evaluate the data to determine if one of the following conditions is met. If these conditions are not met corrective action must be taken. Guidance for troubleshooting is provided in Section 10.2.5. After corrective action the analytical sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If these two CCVs do not meet the criteria, recalibration is required prior to further analysis.

  - If the CCV criteria are exceeded high, indicating a high bias, and the associated samples have non-detects for those analytes, the analytical data may be considered usable. In the absence of instructions otherwise, proceed with analysis.

  - If the CCV criteria are exceeded low, indicating a low bias, analytical results may be reported if those results exceed the project's regulatory decision level. In other words, if the analytical results are sufficiently high to counter the low bias, results may be reported. Consult with the project manager to determine if the exception is allowable for each project.

10.2.5 Troubleshooting

Check the following items in case of calibration failures:

- Loss of sensitivity or unstable ISTD recoveries are usually the result of a leak. Check the union between the GC column and Entech transfer line.

- Loss of sensitivity for individual compounds may be a result of either an active site in a transfer line or a bad trap. Troubleshoot and perform maintenance as necessary.

- Poor chromatography usually requires GC column maintenance, perform as necessary.

- Carryover is usually caused by excessive amounts of analyte introduced to the system. Analyze blanks until the system is cleaned or replace the traps and transfer lines if necessary.

Refer to corporate policy CA-Q-S-005 for additional information of procedures to establish and troubleshoot initial calibration curves.

10.3 Sample Preparation

10.3.1 Post Sampling Canister Pressure Check Procedure

Perform the post-sampling canister pressure check within 1 business day of receipt of canisters in the laboratory so that any problems found are quickly identified and communicated to the client. Record the date and time the post-receipt check is performed is in the analytical record.

To perform the post- sampling canister pressure check:

1) Inspect the condition of equipment for signs of damage. If damage is observed, immediately notify the PM and await further instruction.

2) Record your checks on the Air Canister Post-Sampling Pressure Check Record.

3) Check to see if the sampling FC(s) were returned with the canister(s). If so, check the paperwork (Canister ID Tag, Field Test Data Sheet or Chain of Custody (COC) to determine if the sampling record identifies which FC was used for each canister. If the paperwork does not include this information, record the omission on the post-sampling check record.

   NOTE: The laboratory's sample acceptance policy for air samples in canisters requires that the sampling crew record the ID of the FC used for sample collection on the tag attached to each canister, but the association may also be recorded on the Field Test Data Sheet or a COC. With this information the laboratory can review the history of use of the FC as needed to troubleshoot potential equipment problems. Without the association, the history of use of the FC is unknown. The laboratory strongly recommends that field samplers be instructed to provide this information for each sampling event.

4) Check the pressure of each canister using a digital pressure gauge. Verify that the calibration of the gauge has not expired prior to use. If the calibration sticker indicates calibration checks are past due, notify department management and remove the gauge from service.

   Attach the gauge to the canister inlet. Check for the presence of burr or thread damage when attaching the pressure gauge to the canister inlet. If damage is observed, record the observation on the post-sampling check record

   Open the canister valve and record the pressure reading.

   The pressure should be between -10" Hg to -1"Hg, except for "grab" samples and samples whose sampling time is <200 mL/min, which do not have a return pressure criteria range.

   NOTE: The return canister pressure criteria of -10"Hg to -1"Hg was established based on the recommendations provided in Section V of Appendix I of the Vapor Intrusion Guidance Document prepared by the New Jersey Department of Environmental Protection (NJDEP). These sections of this document describe canister and quality assurance requirements for USEPA Methods TO-15 and TO-17. This document explains that due to recent advances in technology in concentrator units, such as with the Entech concentrators used by the laboratory, it is now possible to remove sample from canisters with a negative pressure of -10"Hg without having to add makeup air. Previous models of concentrators (such as NuTechs) required a pressure of at least -5"Hg. When the return negative pressure of a canister is greater than -10"Hg, the laboratory may need to add makeup air to the canister in order to provide a sufficient amount of sample for analysis. The need for the addition of makeup air depends on the concentrator unit. Some concentrator units used by the laboratory can pull sample without the addition of make-up air when the return negative pressure is up to -15"Hg. The amount of air added to the canister depends on the return pressure reading and will vary with each canister. See section 10.3 for the procedure of the determination of amount of makeup air needed. Except for "grab" samples and samples whose sampling time is set for <200 mL/min, the return pressure of a canister should never be zero negative pressure or a pressure equal to ambient pressure. If it is, the PM must consult with the client and obtain authorization for the laboratory to proceed with analysis.

5) If the return pressure is within range for all samples, photocopy the post-sampling canister pressure check record and attach the record to the screen worksheet.

   If the return pressure is not within range, initiate an anomaly report and perform one of the following actions:

   - Action 1: If the FC was not returned with the canister, attach a copy of the post-sampling pressure check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheet and forward the original anomaly report to the QA department.

   - Action 2: If the FC was with the canister, perform a leak check on the FC gauge and re-check the FC's flow rate as follows. Record all measurements on the original Flow

Controller Set Flow Rate & Leak Check Record. The protective sticker on the back of the FC will provide the page number that corresponds to the logbook record.

To check the flow rate and check the FC gauge for leaks:

1) Remove the stem from the FC.

2) Attach a dust cap where the stem was, and attach the FC to the control gauge/vacuum manifold.

3) Turn the calibrated digital flow meter on and zero the meter.

4) Turn on the vacuum pump and read the vacuum of the control gauge and the FC gauge and record these readings.

5) Turn off the vacuum. Wait ~30 seconds and read the vacuum of the control gauge and the FC gauge and record these readings.

6) The difference between the initial and final readings for the control gauge should be zero. If it is not, there is a leak in the manifold system. Stop work and correct the problem.

7) The difference between the initial and final readings for the FC gauge should be zero. If it is not, there is a leak in the FC assembly. Record the presence of a leak in the record and set aside the FC for service and repair.

8) Remove the dust cap from the FC and re-attach the stem.

9) Attach the flow meter tube to the stem of the FC.

10) Measure the flow rate. The flow rate should be within the ranges specified in the Set Flow Rate Table (See Table 4) for the sampling time requested. If the flow rate is not within range, record the situation on the anomaly report.

11) Attach a photocopy of the Flow Controller Set Flow Rate & Leak Check Record and a photocopy of the post sampling check record to the anomaly report and forward the paperwork to the PM who will notify the client of the situation and request further instruction. The PM will record any decisions made regarding the sample on the anomaly report and return the packet to you. Attach a photocopy of the complete anomaly report to the screen worksheets and forward the original anomaly report to the QA department.

10.3.2 Sample Screening

At the laboratory's discretion unknown samples may be screened prior to initial analysis to determine if the sample requires dilution. Unless otherwise requested by the client the laboratory does not provide screen data with the data package report even when primary dilutions are performed based on the results of the screen analysis.

To prepare a sample for screen analysis connect the sample canister to the autosampler connected to screening instrument and analyze 20 mL of sample. Acquire and evaluate the results. If the results of screen analysis indicate that a target compound is above its upper range of calibration. Calculate a recommended dilution factor (DF) by dividing the concentration of analyte found by 30. Record the recommended DF on the screen worksheet.

NOTE: Samples are screened on a GC/MS instrument that is programmed with the operating conditions given in Section 10.4.1 of this SOP. The calibration is checked weekly with a single point calibration standard at a concentration of 10 ppbv for all target analytes. The calibration is checked more frequently when the results of instrument analysis do not correlate well with the results of the screen analysis.

10.3.3 Sample Dilutions & Pressure Adjustment

Field samples should be diluted prior to initial analysis when the screen results indicate that the concentrations are above calibration range and when the laboratory has sufficient knowledge of the sample (history) to know that the sample will require dilution. Field samples must be reanalyzed at a dilution initial analysis when the concentration of target compounds in initial analysis exceed of the upper range of calibration.

When the return negative pressure of a canister is greater than -15”Hg, make-up air is added to provide sufficient volume of make-up air in order to have an adequate sample volume for analysis. The addition of make-up air is considered a canister dilution.

To dilute the sample:

1) Attach the sample canister to the zero air line equipped with a pressure gauge that reads negative pressure in (“Hg) and positive pressure in (psig).

2) Ensure the valve of the zero air line is closed then open the valve of the sample canister. Record the negative pressure reading in the Canister Dilution Worksheet or on the canister’s tag.

3) Slowly open the valve of the zero air line and fill the canister until canister pressure gauge reads -10”Hg. Do not open the valve to such an extent that the zero air line pressure drops below 15 psig and do not allow the zero air line to reach equilibrium otherwise you will contaminate the zero air line.

4) When the desired pressure is achieved, close the canister valve and the valve on the zero air line; wait 15 seconds.

5) Open the canister valve and record the final pressure reading in psig.

6) Close the canister valve and remove the valve from the zero air line.

7) Record the initial and final pressure readings in the TALS canister dilution tracking module. If the final pressure is below ambient, the “HG reading must be converted to psig by dividing the value by 2 prior to entry into the TALS worksheet.

When the return pressure of a canister is positive, the pressure must be adjusted to near ambient (0"Hg) prior to analysis. To adjust the pressure to ambient, vent the canister to ambient in a fume hood by opening the canister value for ~4-5 seconds, close the valve. For higher pressure canisters, open the valve and listen for a release of air then close the valve when the sound recedes.

If a trip blank is provided, pressurize the trip blank canister to 10 psig. The pressurization of the trip blank is not considered a dilution.

10.3.4 QC Sample Preparation

To prepare the method blank (MB): Fill a clean canister that has never been used to collect environmental samples and has never left the laboratory to 20 psig with zero air. Continue to use this canister as the MB until the pressure of the canister reaches 0 psig, at which time, recharge with zero air to 20 psig and reuse.

To prepare the LCS: Follow the instructions provided in Appendix B for preparation of the working ICV/LCS standard. If an LCSD and replicate precision is requested, the aliquot for the LCSD must be taken from the LCS canister. If an LCSD and duplicate precision is requested, prepare another LCS in a separate canister to serve as the LCSD.

10.4 Sample Analysis

10.4.1 Instrument Operating Conditions

Optimize the GC and MS conditions for compound separation and sensitivity.

The recommended operating conditions are as follows:

Thermal Desorb:	Initial Trap #1 Temperature: -110ºC
	Desorb Temperature from Trap #1 to #2: 0 ºC
	Total Volume Transfer by Mass Flow Controller: 40 mL
	Initial Trap #2 Temperature: -15 ºC
	Desorb Temperature from Trap #2 to #3: 200ºC
	Transfer time 3.5 minutes
	Initial Trap #3 Temperature: -165 ºC
	Injection Trap #3 Temperature: 70ºC
	Injection Time: 1.5 minutes
	Trap #3 Temperature after Injection: -165 ºC
Carrier Gas:	Helium, Ultra High Purity
Cryogenic Focusing Gas:	Liquid Nitrogen
Flow Rate:	~1.5 mL/min
Temperature Program:	Initial Temperature: 40ºC
	Initial Hold Time: 4 minutes
	Ramp1 Rate: 20ºC/min. to 200ºC.
	Ramp 2 Rate: 40ºC/min. to 220ºC
	Final Temperature: 220ºC
	Final Hold Time: 6.5 minutes
Electron Energy:	70 electron volts

Mass Range:	35-265 amu
Scan Time:	≥1 scan per second

These operating conditions may be changed but once the operating conditions are established for initial calibration the same conditions must be used until a new calibration is performed.

10.4.2 Analytical Sequence

An example analytical sequence that includes initial calibration (ICAL) is provided below. When ICAL is not performed, the sequence begins with the tune standard and is followed by the CCV, LCS, LCSD, and method blank. If sufficient time remains in the 24 hours analytical window after initial calibration, QC and field samples may be analyzed without the CCV and the ICV will serve as the LCS for the sequence. The MB, LCS and LSCD must be analyzed at a frequency of every 20 samples or with each analytical sequence whichever is more frequent.

1. Tune Standard (BFB)
2. ICAL
3. ICV
4. CCV
5. LCS (repeat every 20 samples)
6. LCSD (when requested)
7. MB (repeat every 20 samples)
8. Field Samples (including trip blanks)

Attach the canisters to the autosampler inlet in the order of the analytical sequence then initiate the analytical sequence. The autosampler introduces 200 mL of sample volume from each canister to the instrument system and adds 20 mL of the mixed gas standard to each sample.

Acquire the data and evaluate the results to confirm qualitative identification and quantification.

11.0 <u>Calculations / Data Reduction</u>

11.1 Qualitative Identification

The data processing system tentatively identifies target analytes by comparing the retention time of the peaks to the window set around the continuing calibration standard, and searches in that area for the primary ion and up to two secondary ions characteristic of the target analyte.

All tentative identifications made by the computer are reviewed and either accepted or rejected by the primary analyst. The identification made by the system is accepted when the following criteria are met:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the user-created database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some

ions, particularly the molecular ion, are of special importance if a tentative identification is to be made, and should be evaluated even if they are below 10% relative abundance.

- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with laboratory SOP BR-QA-006 *Manual Integration*.

11.2 Quantification of Target Analytes

After a compound has been identified, the data system quantifies the on-column concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

Final results are calculated in TALS.

11.3 Calculations

Analytical results are calculated as follows:

- **Dilution Factor**

$$DF = \frac{V_2}{V_1} x \frac{V_4}{V_3}$$

Where:
V_1 = Pre-Dilution Canister Volume
V_2 = Post-Dilution Canister Volume
V_3 = Sample Amount (mL)
V_4 = Base Sample Amount (200 mL)

- **Relative Response Factor (RRF)**

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

Where:
A_x = Area of the quantitation ion of the analyte
A_{is} = Area of the quantitation ion of the internal standard
C_x = Concentration of analyte in concentration units (ppbv)
C_{is} = Concentration of internal standard in concentration units (ppbv)

- **Percent Relative Standard Deviation (%RSD)**

$$\%RSD = \frac{SD}{Mean} x100$$

Where:
SD = Standard deviation individual response factors
Mean = Average of five response factors

- **Sample Concentration**

$$C_x = \frac{(A_x)(C_{IS})}{(A_{IS})(\overline{RRF})}(DF)$$

Where:
C_x = Compound concentration (ppbv)
C_{IS} = Concentration of associated internal standard (ppbv)
A_{IS} = Area of quantitation ion for associated internal standard
A_x = Area of quantitation ion for compound
DF = Dilution Factor
Mean RRF = Average Relative Response Factor from initial calibration.

- **Unit Conversion from ppbv to ugm3**

$$\text{Analytical Result (ug/m3)} = \text{Result(ppbv)} \times \left(\frac{mw}{24.45}\right)$$

Where:
mw = molecular Weight

Example:
Benzene Result = 56 ppbv
Benzene mw = 78.108

$$\text{Analytical Result (ug/m3)} = 56\ \text{ppbv} \times \left(\frac{78.108}{24.45}\right)$$

Result(ug/m3) = 178.9 ug/m3 reported as 180 ug/m3

- **Percent Recovery (%R)**

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Where:

C_s = Concentration of the spiked sample (ppbv)
C_n = Nominal concentration of spike added (ppbv)

- **Precision (%RPD)**

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

Where:
C_1 = Measured concentration of the first sample aliquot
C_2 = Measured concentration of the second sample aliquot

11.4 Data Review

11.4.1 Primary Review (Performed by Primary Analyst)

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the NCM is suspected, which action (if any) was taken and why and the outcome of the action taken.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Set the batch to 1st level review.

Record your review on the data review checklist.

11.4.2 Secondary Review (Performed by Peer Reviewer)

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP are were approved and are properly documented.

Spot-check 15% of samples in the batch to verify quantitative and qualitative identification.

If manual integrations were performed:

- Review each manual integration to verify that the integration is consistent and compliant with the requirements specified in laboratory SOP BR-QA-005.

- Check to ensure an appropriate technical reason code is provided for each manual integration. Acceptable technical reason codes are provided in laboratory SOP BR-QA-005.

- Generate a “before” and “after” chromatogram for every manual integration performed on an instrument performance check standard (Tune, ICAL, ICV, CCV), QC sample (MB, LCS) and for any manual integration performed on any surrogate or internal standard in any field sample.

- Generate the Manual Integration Summary Report. Document your review of manual integrations on the summary report and obtain any review signatures of integrations performed during secondary review as required.

  If the reviewer disagrees with the integration performed by the primary analyst, the secondary data reviewer should not change the integration. Instead, he/she should consult with the primary analyst that performed the integration and both the reviewer and the primary analyst should agree the integration should be changed. If consensus between the primary analyst and the peer reviewer cannot be achieved; both should consult with the Technical Manager or department management for resolution. Any changes to the integration should be performed by the primary analyst. If it is necessary for the secondary reviewer to perform the manual integration because the primary analyst is out of the office; the integration made by the peer reviewer must be reviewed by another peer reviewer or by department management to verify the integration was performed and documented in compliance to SOP BR-QA-005. If the original analyst that performed the integration is out of the office, the data reviewer may consult with the Department Manager (DM), Department Supervisor (DS) or the Technical Manager (TM) to verify the change he/she thinks is needed is warranted and should be made.

Verify that the performance criteria for the QC items listed in Table 1 were met. If the results do not fall within the established limits verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review.

Run the QC checker and fix any problems found. Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

Record second level review on the data review checklist.

11.5 Data Reporting

Data reporting and creation of the data deliverable is performed by TALS using the formatters set by the project manager during project initiation.

Electronic and hardcopy data are maintained as described in laboratory SOP BR-QA-014 Laboratory Records.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005,

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out:

- None

15.0 References / Cross-References

- EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-001 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- LaboratorySOP BR-QA-006 Procedures & Documentation Requirements for Manual Integration
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

Not Applicable.

17.0 Attachments

- Table 1: Target Compound List, RL, Internal Standard and Ion Assignments
- Table 2: Ion Abundance Criteria (BFB)
- Table 3: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables

18.0 Revision History

BR-AT-004r8, Revision 8:

- Title Page: Updated approval signatures
- All sections: Added procedure for ethanol
- Section 10: Changed calibration groups

Revision 7:
Section 11.4.1: Expanded on discussion of carry over.
Appendix C: Removed.
Appendix D: Removed.
Appendix E: Removed.

Table 1: Routine Compound List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dichlorodifluoromethane	75-71-8	0.5	5	85	87		1	B
Freon-22	75-45-6	0.5	5	51	67	69	1	B
1,2-Dichlorotetrafluoroethane	76-14-2	0.2	2	85	135	87	1	A
Chloromethane	74-87-3	0.5	5	50	52		1	B
n-Butane	106-97-8	0.5	5	43	41	58	1	B
Vinyl Chloride	75-01-4	0.04	0.40	62	64		1	D
1,3-Butadiene	106-99-0	0.5	5	54			1	B
Bromomethane	74-83-9	0.2	2	94	96		1	A
Chloroethane	75-00-3	0.5	5	64	66		1	B
Isopentane	78-78-4	0.2	2	43	57	56	1	A
Bromoethene (Vinyl Bromide)	593-60-2	0.2	2	106	108	81	1	A
Trichlorofluoromethane	75-69-4	0.2	2	101	103		1	A
Pentane	109-66-0	0.5	5	43	57	72	1	B
EthylEther	60-29-7	0.2	2	59	45	74	1	A
Acrolein	107-02-8	5	50	56	55	37	1	C
Freon TF	76-13-1	0.2	2	101	151	103	1	A
1,1-Dichloroethene	75-35-4	0.2	2	96	61	63	1	A
Acetone	67-64-1	5	50	43	58		1	C
Isopropyl Alcohol	67-63-0	5	50	45	43		1	C
Carbon Disulfide	75-15-0	0.5	5	76			1	B
3-Chloropropene (Allyl Chloride)	107-05-1	0.5	5	41	76		1	B
Acetonitrile	75-05-8	5	50	41	40	39	1	C
Methylene Chloride	75-09-2	0.5	5	49	84	86	1	B
tert-Butyl Alcohol	75-65-0	5	50	59	41	43	1	C
Methyl tert-Butyl Ether	1634-04-4	0.5	5	73	43		1	B
trans-1,2-Dichloroethene	156-60-5	0.2	2	61	96		1	A
n-Hexane	110-54-3	0.5	5	57	86		1	B
1,1-Dichloroethane	75-34-3	0.2	2	63	65	83	1	A
Methyl Ethyl Ketone	78-93-3	0.5	5	72	43		1	B
cis-1,2-Dichloroethene	156-59-2	0.2	2	96	98		1	A
Tetrahydrofuran	109-99-9	5	50	42	72		2	C
Chloroform	67-66-3	0.2	2	83	85		1	A
1,1,1-Trichloroethane	71-55-6	0.2	2	97	99	61	2	A
Cyclohexane	110-82-7	0.2	2	84	56		2	A
Carbon Tetrachloride	56-23-5	0.2	2	117	119		2	A
2,2,4-Trimethylpentane	540-84-1	0.2	2	57	41	43	2	A
1,2-Dichloroethene (total)	540-59-0	0.2	2	61	96		1	A
Benzene	71-43-2	0.2	2	78	77		2	A
1,2-Dichloroethane	107-06-2	0.2	2	62	98		2	A
n-Heptane	142-82-5	0.2	2	43	71		2	A
Trichloroethene	79-01-6	0.04	0.40	95	130	132	2	D
Methyl Methacrylate	80-62-6	0.5	5	69	41	39	2	B
1,2-Dichloropropane	78-87-5	0.2	2	63	41		2	A
1,4-Dioxane	123-91-1	5	50	88	58		2	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dibromomethane	74-95-3	0.2	2	174	93	172	2	A
Bromodichloromethane	75-27-4	0.2	2	83	85		2	A
cis-1,3-Dichloropropene	10061-01-5	0.2	2	75	110		2	A
Methyl Isobutyl Ketone	108-10-1	0.5	5	43	58		2	B
n-Octane	111-65-9	0.2	2	43	57	114	2	A
Toluene	108-88-3	0.2	2	92	91		3	A
trans-1,3-Dichloropropene	10061-02-6	0.2	2	75	110		2	A
1,1,2-Trichloroethane	79-00-5	0.2	2	83	97	85	3	A
Tetrachloroethene	127-18-4	0.04	0.40	166	168	129	3	D
Methyl Butyl Ketone	591-78-6	0.5	5	43	58		3	B
Dibromochloromethane	124-48-1	0.2	2	129	127		3	A
1,2-Dibromoethane	106-93-4	0.2	2	107	109		3	A
Nonane	111-84-2	0.2	2	57	71	128	3	A
Chlorobenzene	108-90-7	0.2	2	112	77	114	3	A
Ethylbenzene	100-41-4	0.2	2	91	106		3	A
Xylene (m,p)	1330-20-7	0.5	5	106	91		3	A
Xylene (o)	95-47-6	0.2	2	106	91		3	A
Styrene	100-42-5	0.2	2	104	78		3	A
Bromoform	75-25-2	0.2	2	173	175	171	3	A
Cumene	98-82-8	0.2	2	105	120	77	3	A
1,1,2,2-Tetrachloroethane	79-34-5	0.2	2	83	131	85	3	A
Xylene (total)	1330-20-7	0.2	2	106	91		3	A
n-Decane	124-18-5	0.5	5	57	71	142	3	B
n-Propylbenzene	103-65-1	0.2	2	91	120	92	3	A
1,2,3-Trichoropropane	96-18-4	0.5	5	75	110	112	3	B
4-Ethyltoluene	622-96-8	0.2	2	105	120		3	A
1,3,5-Trimethylbenzene	108-67-8	0.2	2	105	120		3	A
2-Chlorotoluene	95-49-8	0.2	2	91	63		3	A
tert-Butylbenzene	98-06-6	0.2	2	119	91	134	3	A
1,2,4-Trimethylbenzene	95-63-6	0.2	2	105	120		3	A
sec-Butylbenzene	135-98-8	0.2	2	105	134	91	3	A
4-Isopropyltoluene	99-87-6	0.2	2	119	134	91	3	A
1,3-Dichlorobenzene	541-73-1	0.2	2	146	111	148	3	A
1,4-Dichlorobenzene	106-46-7	0.2	2	146	111	148	3	A
n-Undecane	1120-21-4	5	50	57	71	156	3	C
Benzyl Chloride	100-44-7	0.2	2	91	126	65	3	A
n-Butylbenzene	104-51-8	0.2	2	91	134	92	3	A
1,2-Dichlorobenzene	95-50-1	0.2	2	146	111	148	3	A
n-Dodecane	112-40-3	5	50	57	71	170	3	C
1,2,4-Trichlorobenzene	120-82-1	0.5	5	180	182		3	B
1,3-Hexachlorobutadiene	87-68-3	0.2	2	225	223		3	A
Naphthalene	91-20-3	0.5	5	128			3	B
1,2,3-Trichlorobenzene	87-61-6	0.2	2	180	182	145	3	A
Propylene	115-07-1	5	50	41	42	39	1	C
Vinyl Acetate	108-05-4	5	50	43	86		1	C

Analyte	CAS No.	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Ethyl Acetate	141-78-6	5	50	43	74		1	C
Ethanol	64-17-5	5	50	46	45		1	E
Bromochloromethane	74-97-5	NA	NA	128	49	130	1	NA
1,4-Difluorobenzene	540-36-3	NA	NA	114			2	NA
Chlorobenzene-d5	3114-55-4	NA	NA	117			3	NA

Table 2: Tune Standard Criteria

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of mass 95
75	30.0 to 66.0 percent of mass 95
95	Base Peak, 100 percent relative abundance
96	5.0 to 9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of mass 95
175	4.0 to 9.0 percent of mass 174
176	93.0 to 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

Table 3: TO15 QC Summary & Recommended Corrective Action

QC Check	Frequency	Acceptance Critera	Recommended Corrective Action
Tune Standard	Prior to calibration and every 24 hours	See Table 2	Correct Problem. Reanalyze. No samples may be analyzed without a valid tune.
ICAL	Prior to sample analysis and when CCV fails	RSD for each analyte ≤ 30% with 2 exceptions up to 40%	Correct problem and repeat calibration
ICV	Once after each ICAL	%R for all analytes within 70-130	Correct Problem. Reanalyze, re-make, re-verify & re-analyze. If that fails, re-make all standards and repeat calibration.
Retention Time Window	Once per ICAL	NA	NA
RRT	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct Problem. Repeat ICAL
CCV	Daily before sample analysis after tune standard	%D ≤ 30	Correct Problem. Reanalyze once. If that fails, see section 10.2.5 for instruction.
LCS	Each batch or every 20 samples, whichever is sooner.	%R for all analytes within 70-130	Reanalyze LCS, re-prep and reanalyze LCS and all associated samples if sufficient sample volume is available. If corrective action not successful, initiate nonconformance report and qualify sample results.
LCSD	Per Client Request	RPD ≤ 25	Reanalyze LCSD, re-prep and reanalyze LCSD and all associated samples if sufficient sample volume is available. If corrective action is not successful, initiate nonconformance report and qualify sample results.
Method Blank	Each batch or every 20 samples, whichever is sooner.	No analytes detected above RL	Reanalyze along with associated samples, unless detects for same compounds found in blank are greater than 10X the concentration found in the blank.
Internal Standard	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Inspect system for malfunction. Reanalyze samples. Qualify data.
Sample Duplicate	Per Client Request	RPD ≤ 25	Consult with PM. Reanalyze or qualify data.

Appendix A: Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: 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) components which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: Environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): An analytical standard gas mixture containing all target analytes and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8℃) or liquid argon (bp -185.7℃).

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV): An analytical standard mixture containing all target analytes and internal standard compounds that are prepared from a source independent of the source of the initial calibration standards. The purpose of the ICV is to verify that the initial calibration is in control.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Internal Standards (IS): Non-target analytes that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). IS are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.

Laboratory Control Sample (LCS) – A QC sample of known composition spiked with analytes of interest. The LCS evaluates method performance and ability to successfully recover target analytes from a clean matrix. LCS recovery is typically expressed as percent recovery and provides a measure of accuracy. A LCSD is a duplicate LCS prepared and analyzed from a separate canister to provide a measure of replicate precision.

Method Blank (MB): A canister of humidified ultra pure zero air that is treated exactly as a sample. The MBLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a <u>range</u> where qualitative detection occurs. Quantitative results are not produced in this range.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Gas Mixture: A Commercially purchased concentrated gas mixture containing one or more method analytes

Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory SOP BR-QA-002 *Standard Preparation* for further guidance on the preparation of standard solutions.

Prepare all standards using the McMillan Company 80SD mass flow controller. Prepare the standard in zero air, demonstrated to be analyte free. Store the standard at ambient temperature. Unless otherwise specified, assign an expiration date of 30 days from date of preparation unless the parent standard expires earlier, in which case, use the earliest expiration date.

Intermediate Calibration Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Custom Calibration Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15 L Summa Canister Expiration Period 3months
This standard contains all the target analytes listed in table 1.

Working Calibration Standards

Parent Standard	Calibration Standard	Parent Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Cal Standard 20 ppbv	Cal Standard 0.2 ppbv	20	155	15.46	0.2
Cal Standard 20 ppbv	Cal Standard 0.5 ppbv	20	386	15.46	0.5
Intermediate Calibration Standard	Cal Standard 5 ppbv	200	386	15.46	5
Intermediate Calibration Standard	Cal Standard 10 ppbv	200	773	15.46	10
Intermediate Calibration Standard	Cal Standard 15 ppbv	200	1160	15.46	15
Intermediate Calibration Standard	Cal Standard 20 ppbv	200	1546	15.46	20
Intermediate Calibration Standard	Cal Standard 40 ppbv	200	3092	15.46	40

Prepare in 6 L Summa Canister Expiration Period 3 months
Each calibration standard contains all the analytes listed in table 1 at the above concentrations.

Initial Calibration Levels

Calibration Level	Working Calibration Standard	Volume Analyzed (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.2 ppbv	200	0.2
Calibration Level 2	Cal Standard 0.5 ppbv	200	0.5
Calibration Level 3	Cal Standard 5 ppbv	200	5
Calibration Level 4	Cal Standard 10 ppbv	200	10
Calibration Level 5	Cal Standard 15 ppbv	200	15
Calibration Level 6	Cal Standard 20 ppbv	200	20
Calibration Level 7	Cal Standard 40 ppbv	200	40
Calibration Level 8	Cal Standard 0.2 ppbv	40	0.04

Prepare in 6L Summa Canister

Intermediate ICV/LCS Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)

ICV Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Working ICV/LCS Standard

Parent Standard	Calibration Standard	Stock Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Intermediate ICV/LCS Standard	ICV Standard 10 ppbv	200	773	15.46	10

Prepare in 6L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in table 1.

Intermediate Ethanol Calibration Standard at 500ppbv/v

1) Fill a 44 ml VOA vial with VOA free water. Remove 197ul of water from the vial.
2) Add 197 ul of >99.5% Ethanol neat material
3) Cap and shake/roll vial for 1 minute
4) Inject 10ul of the prepared water/ethanol mix into a fully evacuated 15 liter summa canister
5) Pump the syringe plunger 5 times to insure complete transfer of material
6) Immediately fill the canister to 22 psig with zero air.

Calibration Level	Working Calibration Standard	Volume added (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.5 ppbv	124	5
Calibration Level 2	Cal Standard 5.0 ppbv	309	10
Calibration Level 3	Cal Standard 10ppbv	464	15
Calibration Level 4	Cal Standard 15 ppbv	618	20
Calibration Level 5	Cal Standard 20 ppbv	1237	40
Calibration Level 6	Cal Standard 40 ppbv	3092	100

TITLE: Gas Chromatography - Semi-Volatiles Pesticides by SW-846 Method 8081A and 8081B

Approvals (Signature/Date):

Patti Gibson 9/24/13 Patti Gibson — Date GC Chemist	
9/24/13 Gary L. Rynkar — Date Supervisor GC Extr./HPLC	9/24/13 Chris Hoham — Date Env. Health & Safety Coor.
Terese A. Preston 9/24/13 Terese A. Preston — Date Quality Assurance Manager	Michael J. Healy 9/24/13 Michael J. Healy — Date Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the procedure used to determine the concentration of chlorinated pesticides by gas chromatography (GC) in assorted matrices using SW-846 Methods 8000B/8081A, 8000B/8081B and 8000C/8081B.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Table 1 provides a list of compounds analyzed by this method and their reporting limits.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

This method provides the GC conditions and necessary standardization procedures for the detection of parts per billion (ppb) levels of certain chlorinated pesticides. Prior to this method, the appropriate extraction must be performed depending upon the sample matrix (refer to Section 7.3.1). Confirmation must be obtained for all positive results and this may be accomplished by using retention time data from a dissimilar column.

2.0 INTERFERENCES

2.1 Phthalates

- Interference's by phthalate esters can pose a major problem in pesticide analysis. These compounds generally appear in the chromatogram as large late-eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps especially when solvent-wetted surfaces are handled. Interference from phthalates can best be minimized by avoiding contact with any plastic materials.

2.2 Co-extractions

- Co-extracted interferences will vary considerably from sample to sample. Clean-ups such as a florisil clean up, sulfur clean up, and GPC clean up are available to help eliminate or lessen interferences (refer to UP-SP-003).

2.3 Glassware

- All glassware/equipment used in the preparation of standards (volumetric flasks, pipettes, beakers, eppendorf tips) are rinsed well with the appropriate solvent and allowed to dry before use. After completion of standard preparation, unused amounts of standards are properly disposed of, and all glassware/equipment rinsed well with the appropriate solvent. Rinse/excess solvent is properly collected and disposed of. These procedures should eliminate positive interferences from these sources.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Pesticides and PCBs are a bio-accumulating carcinogen. Extreme care should be taken when working with this material.
- The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- There are areas of high voltage in the GC. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- ECDs contain a radioactive source. Special regulations apply.
- Employees will read the MSDSs for the materials used in this method before handling or using the material.
- Special regulations apply to Pesticide/PCB handling and disposal.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

4.1 Supplies

- Autosampler vials (2 mL) with Teflon-lined caps
- Pipets - Class A volumetric
- Transfer pipets
- Volumetric flasks - Class A

4.2 Gas Chromatographs

GCs used for this analysis are equipped with dual electron capture detectors (ECDs) (Ni_{63}). Each GC is equipped with an autosampler and uses Total Chrom for data acquisition and Chrom for processing data.

4.3 Analytical Columns

- Column 1/signal 0 – Restek Rtx-CLPesticides (Rtx-CLP1), 0.53 mm ID, 30-m length 0.5 micron film thickness (or equivalent).
- Column 2/signal 1 – Restek Rtx-CLPesticides2 (Rtx-CLP2) 0.53 mm ID, 30-m length 0.4 micron film thickness (or equivalent).

Signal 0 is designated as the primary column in TALs and signal 1 is designated as the secondary column in TALs. Additional guidance can be found in Attachment 5, the corporate SOP CA-T-P-003, *Reporting Results for Methods That Require Second-Column Confirmation*. Similar columns can be used if all QC criteria can be met.

5.0 REAGENTS AND STANDARDS

5.1 Solvents

Hexane, Acetone, Methanol - Pesticide quality or better.

5.2 Quality Control (QC) Solutions

Stock standards are purchased as solutions generally from Ultra or Restek.

Label / Storage Information: The preparation of all standards and QC solutions must be properly documented. A unique reagent code is created in the TALs reagent module. The information includes the name of the reagent, the concentration of the reagent, the date prepared, expiration date and the analyst who prepared the reagent. The reagent label used to label each reagent bottle contains the unique TALs code for that reagent bottle, its description, expiration date, prepped/received date, solvent used, prepared by and date created. The label also contains a bar code for scanning into the Chrom worklists, which then transfer to the TALs AD batches upon upload.

Standards must be stored in glass containers with Teflon-lined lids at 4 $\pm$ 2°C, protected from the light. Stock standards must be replaced after 1 year, or sooner if comparison with a check standard indicates a problem.

5.2.1 Stock Calibration Standards

Stock Standards are generally purchased from Restek as a certified material.

The concentrations of the stock solutions of INDAB, Toxaphene and Chlordane result in the need of an intermediate standard for these compounds. The resulting concentrations of the intermediate solution for INDAB, Toxaphene and Chlordane are 2, 100 and 10.0 ng/uL, respectively. All standards are prepared in hexane.

The surrogate compounds 2,4,5,6-Tetrachloro-m-xylene (TCX) / Decachlorobiphenyl (DCB) are purchased as a mix. They are diluted to a concentration of 2.0 ng/uL in hexane.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.1.1 Working Calibration Standards

Working calibration standards are prepared in hexane from the stock solutions at 5 or 6 levels (Table 2).

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.2 Stock Spike Solution: 200 ug/mL

A stock spike solution containing all routine single response pesticide compounds at 2000 ug/mL is purchased from Restek (a source different than the calibration standards).

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.2.1 Working Spike Solution: 0.4 ug/mL

A 0.1-mL aliquot of the Stock Spike Solution is diluted into 50mLs of methanol (1/500 dilution) making a parent solution at concentration of 4 ug/mL. This parent solution is given to the extraction group, where they make a 1/10 dilution by taking 10mLs and diluting it into 100mLs of methanol resulting in a working spike solution at a concentration of 0.40 ug/mL.

1.0 mL of this solution is then added to the Laboratory Control Sample (LCS), matrix spike and matrix spike duplicate (MS/MSD). The final volume of the extract is 10mLs, resulting in an extract concentration of 0.04 ng/uL.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.3 Surrogate Stock Solution: 200 ug/mL

Surrogates are added to each sample, method blank (MB), LCS and MS/MSD prior to extraction. A single solution containing the surrogates (DCB and TCX) is purchased from a vendor (Restek) at a concentration of 200 ug/mL.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.3.1 Working Surrogate Solution: 0.4 ug/mL

A 2mL aliquot of the Surrogate Stock Solution is diluted into 100mLs of acetone (1/50 dilution) making a parent solution at concentration of 4.0 ug/mL. This parent solution is given to the extraction group, where they make a 1/10 dilution by taking 10mLs and diluting it into 100mLs of acetone resulting in a working spike solution at a concentration of 0.40 ug/mL. This source is a source separate from calibration standards.

1mL of this solution is then added to every sample, MB, LCS and MS/MSD prior to extraction. The final volume of the extract is 10mLs, resulting in an extract concentration of 0.04 ng/uL.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.4 Continuing Calibration Verification (CCV) Standard

The continuing calibration verification standard for INDAB corresponds to level 4 of the calibration curve (Table 2). The CCV standard is prepared independently of the initial calibration standards. Technical Chlordane (when needed) and Toxaphene correspond to level 3 of the calibration curve along with the single response pesticides every 10th injection thereafter or 12 hour sequence.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.5 Evaluation B Standard (EVAL B)

The degradation of Endrin and 4,4'-DDT is monitored by analyzing an evaluation standard which contains Endrin at 0.01 ng/uL and 4,4'-DDT at 0.02 ng/uL.

- Label / Storage Information: Refer to Section 5.2
- Life of Standard: This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

6.0 CALIBRATION (NON-DAILY)

6.1 External Calibration

For each analyte of interest, prepare the calibration standards at a minimum of 5 concentration levels by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane (refer to Section 5.2.1.1). One of the external standards should be at the concentration equivalent to or below, the reporting limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

Inject each calibration standard using the technique that will be used to introduce the actual samples into the GC. If the percent relative standard deviation (%RSD) of the calibration factor is ≤ 20% for the linearity over the working range, linearity through the origin can be assumed, and the average calibration factor (CF) may be used for calculations. If an individual RSD is >20%, but the average across all analytes is <20%, analysis may continue, unless state or program criteria prohibit the use of the Grand Mean approach. Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the calibration curve. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed. Note: Care should be taken when using this approach because if hits are detected in the sample, the result may be biased if it's individual RSD was >20%. Alternatively, if the correlation coefficient is >0.99, linear regression may be used.

$$\text{Calibration Factor} = \frac{\text{Height or Area of Peak}}{\text{Mass Injected (in nanograms)}}$$

*For multi-response pesticides, use the individual height or area of all peaks to determine calibration factor and then average used for quantitation.

The working calibration curve or average CF must be verified on each working day and after every 10 injections[2] by injecting a CCV standard. If the response for any analyte varies from the predicted response by more than ±15%, the average % difference across all analytes may be used. Care should be taken when using this approach because if hits are detected in the sample, the result may be biased if its individual %D was >15%. If the average response for all analytes is ±15%, then calibration has been verified. If the average % difference is >15%, corrective actions must be taken in order to bring the instrument back into control, or a new calibration curve must be analyzed.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5 pt. Curve minimum)	≤ 20% RSD (grand mean <20%)[1]
Second Source (SSV)	After initial calibration	± 15% Pred. Resp. (8081A); ± 20% (8081B)
Retention Time Windows	Established with initial calibration	3 X Standard Deviation
Cont. Cal. Verif. (CCV)	every 10 samples [2] or 12 hours	15% D of avg. CF's or predicted response (8081A)[1] 20% D of avg. CF's or predicted response (8081B)

[1] Note: Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the ICAL and CCV. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed.
[2]Suggested to minimize reruns – The method requires that the CCV be analyzed after every 20 samples.

6.2 Retention Time (RT) Windows

Before establishing windows, make sure the GC system is within optimum operating conditions. Make 3-injections of all the single component standard mixtures and multi-response compounds throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in RT windows that are too tight, the minimum RT window criteria is ± 0.03 min.

Calculate the standard deviation of the 3-absolute RTs for each single component standard. For multi-response compounds, choose one major peak from the envelope and calculate the standard deviation of the 3 RTs for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

Plus or minus 3X the standard deviation of the absolute RTs for each standard will be used to define the RT window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multi-response compounds, the analyst should use the RT window but should primarily rely on pattern recognition.

If the standard deviation of the RTs for a target compound is 0.000 (no difference between the 3 RTs), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. The laboratory must calculate RT windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory and available for review.

The RT windows for the analytical sequence are established for all pesticides, multi-response compounds, and surrogates. All subsequent standards must fall within those established windows. RT windows can be updated daily with the analysis of the beginning calibration verification standards. Most often RT windows are updated at the onset of a continuing calibration sequence or after maintenance has been performed.

7.0 PROCEDURE

7.1 Quality Control Checks

The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4,5]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for the MS/MSD is randomly selected by the extraction lab, unless specifically designated by the client on the COC.
[3] Analytical and QC samples (MB, LCS, MS/MDS)
[4] Statistical control limits are updated annually and are updated into TALs (LIMS).
[5] State specific or program specific acceptance criteria may be required, check the Project/Job notes in LIMs to determine if any custom limits are applicable. Ex. For the state of South Carolina, the LCS results must meet a 70 – 130% recovery window.

7.2 Sample Preservation and Storage

All samples should be collected in glass containers with a Teflon-lined lid. If the lid does not have a Teflon liner, aluminum foil should be placed between the jar and lid (dull side facing sample). All samples should be refrigerated up until extraction.

Matrix	Holding Time To Extraction (VTS[1])	Holding Time to Analysis (VTS)	Preservative[2]
Soil/Sediment	14 days (VTS)	\	\
Water	7 days (VTS)	40 days after extraction	Cool, 4 ± 2°C
Waste/Oil	14 days (VTS)	/	/

[1] VTS = verified time of sampling
[2] Prior to and after extraction.

7.3 Sample Preparation

7.3.1 Extraction Procedure

The matrix determines which extraction procedure to follow. Waters are extracted by SW-846 Method 3510C (UP-SP-3510); soils are by Method 3550B or 3541 (UP-SP-3550 or -3541); and organic waste and oily matrices are by Method 3580A (UP-SP-3580).

7.3.2 Clean-up Procedure

Various clean-up procedures are available if interference is present. Florisil, mercury and GPC are most commonly used (UP-SP-003).

7.4 Calibration / Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

7.4.1 Calibration Evaluation and Acceptance Criteria

Refer to the TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (Attachment 1)

7.5 Gas Chromatographic Analysis

7.5.1 GC Conditions - Megabor

- Carrier Gas (Hydrogen) 3 - 10 mL/min
- Make up Gas (Nitrogen) > 60 mL/min
- Column Temp Program 110-320°C (based on elution profile)
- Ramp 6-30°C/min
- Injector Temp 240°C
- Detector Temp 300°C

NOTE: Subject to change based upon instrument performance.

The oven temperature and the carrier gas flow may vary slightly from instrument to instrument and from column to column. The criterion that must be met to determine the actual flow and temperature is: resolution criteria from determinative method must be met

Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column may need to be primed or deactivated by injecting a 0.5-ppb pesticide standard which is 20X the mid-level standard of 0.04 ppb - blanks are then analyzed prior to standardization to ensure the absence of cross-contamination. Inject this prior to beginning initial or daily calibration.

4,4'-DDT and Endrin are easily degraded if the injection port or front of the column is dirty. This is the result of buildup of high boiling residue from sample injection. Breakdown is monitored throughout the analytical sequence with the analysis of and evaluation standard containing Endrin and 4,4'-DDT. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and Endrin (Endrin ketone and Endrin aldehyde). If degradation of either 4,4'-DDT or Endrin is $\geq$ 15%, take corrective action before proceeding with instrument calibration. Breakdown should be checked at the beginning of each 12-hour analytical sequence. Refer to Section 9.8 for the breakdown calculations.

Following the initial calibration the analysis sequence begins with continuing instrument calibration followed by sample extracts. Continuing calibration verification standards for all compounds of interest are run before and after every 10 samples*. The sequence must stop when qualitative and/or quantitative QC criteria are exceeded.

***Suggested** - Must be run after 20 samples or within 12 hours of beginning calibration verification standards. Refer to Table 3 for analytical sequence of quantitation and confirmation.

The calibration factor for each analyte to be quantitated should not exceed a 15% difference when compared to average CF from the initial calibration. If the response for any analyte varies from the predicted response by more than $\pm$ 15%, then the average % difference ($\pm$ 15) across all analytes may be used (8081A), unless state, method (8081B) or program criteria prohibit the use of the Grand Mean approach. Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the calibration curve. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary. The instrument must be within control before proceeding with sample analysis. All samples that were injected after the standard exceeding the criteria must be re-injected. If routine maintenance does not return the instrument back into control based on the last initial calibration, verify that proper maintenance has been performed and if it is still out of control, start a new initial calibration.

If the responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Over lapping peaks are not always evident when peaks are off scale. Computer reproductions of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

If peak detection is prevented by the presence of interferences, further clean-ups may be required.

7.6 Preventive Maintenance

- The septa should be changed between each analysis sequence. The number of injections made plays a part in how frequently they should be changed. Generally, not more than 125-150 injections should be made without changing the septum.
- The entire system should be periodically checked for leaks and frayed wires.
- Leak tests must be performed every six months on every ECD.
- Inspect injection end of column for particulates and/or cracks.
- Check press connectors. (If applicable)
- Condition of the column - upper end of column temperature 285°C for 1 - 2 hours.
- Remove 1-6 inches from the injector and guard column. (If applicable)
- Make sure septum doesn't need to be changed.

Refer to the instrument manuals if problems are encountered which can not be solved by routine maintenance.

7.6.1 Manual Integration Policy

In each case where manual integration has taken place, Chrom will apply the operator's name and date and reason for the manual integration to the electronic file. Manual integrations shall follow the TestAmerica Corporate SOP for Manual Integrations (CA-Q-S-002) located in Attachment 4. An example of one such integration and documentation is included in the attachment. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

Manual integrations are most often performed for the following reasons.

Peak Not Integrated	Split Peak
Sample Matrix Interference	Assign Peak
Column Bleed	Baseline Smoothing
Instrument Noise	Isomers
Peak Tail	Shouldering
Incomplete Integration	Other
Wrong Peak	

A peer review is performed on all manual integrations. A pdf of all the raw data including the before and after manual integrations are automatically imported into the TALs AD batch from Chrom. A summary of the MIs are generated in the TALs deliverable and is include in the data summary portion of the data package. The pdf of the MIs are automatically included in the Level 4 deliverable.

7.7 Documentation

7.7.1 Instrument Run-Logs

The analysis of samples and standards is documented within each instrument-specific run log (Attachment 2), and must be completed for each day's analysis.

7.7.2 Traceability of Standards

Upon receipt or preparation, each standard is entered into LIMS and is issued a unique ID# based upon the type and sequential order in which the item was received. Further information includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., **XXXX** Stock Solution – LCS/MS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements, relevant EPA methodologies and in accordance with the TestAmerica corporate procedure CA-T-P-003, *Reporting Results for Methods that Require Second-Column Confirmation* (Attachment 6). The analysts transfer the data into the TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 3) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 At least one MB and LCS will be included in each laboratory lot of 20 or fewer samples. The MB will be examined to determine if contamination is being introduced in the laboratory. The MB, LCS and MS/MSD must be carried through all stages of the sample preparation, cleanup procedures and measurement steps. The batch QC that has gone through the cleanup procedure will be analyzed with the samples and uploaded into TALs. Note: If only a portion of the samples in the batch required the cleanup procedure, the batch QC will be set to a status of 'Acceptable'. This data will be available to the client upon request. The QA department generates in-house statistical limits using the TALs Control Charting Module on an annual basis.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Precision will be measured by the reproducibility of the MSs and will be calculated as Relative Percent Difference (RPD). If MSs were not analyzed, reproducibility will be measured using the LCS/LCD. Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 Surrogate compounds will be added to every analytical and QC sample to measure the performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.1.5 Each time an analytical sequence is started, the first pesticide standard must be evaluated to determine if the chromatographic system is operating properly. The analyst should consider: 'do the peaks look normal?' and 'is the response obtained comparable to the response from previous calibrations?'. Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc..

8.1.6 Before analysis of any samples, the analyst should demonstrate, through the analysis of a MB that interference from the analytical system, glassware and reagents are under control.

8.1.7 If any changes are made to the chromatographic system, recalibration of the system must take place.

8.1.8 The laboratory must maintain records to document the quality of the data generated. When results of the LCS indicate irregular method performance, a quality control check standard should be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Corrective Actions

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources when determining the action to be taken. The out of control situation may or may not be caused by more than one problem. The analyst should seek the help of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation and the required corrective action. The analysis must not be resumed until the source of the problem and an in control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the section manager, supervisor, QA Personnel or the lab manager.

Listed below are steps that MUST be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a Non-Conformance Memo (NCM);
- document in the NCM that an in control situation has been achieved; and
- receive approval of the section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

Whenever a problem exists that needs to be addressed a NCM is written. It is filed with the report discussing the actions taken to correct and document the problem. The analyst and their section manager decide what actions to take with input from to do with this problem, whether it is analytical, sampling, or matrix interference.

Throughout Sections 6.0 & 7.0 numerous criteria are describe which must be met to meet analytical requirements.

Listed below are some suggested courses of action that may be taken to correct out of control situations that may occur with the procedure.

8.2.1 Initial Calibration

If the initial calibration criteria described in Section 6.1 is not met, first verify all calculations and concentrations are correct. If still out of control, re-analyze the curve. If the re-analysis does not meet the criteria new stock and/or working standards may be needed. Prior to running a new calibration curve or preparing new standards, ensure proper maintenance has been performed.

8.2.2 Continuing Calibration Verification (ICV) (INDAB and Multi-Component Standards)

If through out the analytical sequence any CFs for any of the analytes exceeds the 15% difference limit for 8081A or the 20% difference limit for 8081B, corrective action must take place. First, try re-injecting the standard. If that does not make a difference, refer to Section 7.5.5 for recommended maintenance. If the CF's are still unacceptable, a new analytical sequence should be started. All samples that were injected after the standard that last met QC criteria must be re-injected.

Repeat the standard to verify proper preparation. If still non-compliant, prepare a new standard from the original stock, recalibrate with a new standard curve, or prepare new stock and/or working standards.

8.2.3 Laboratory Control Sample (LCS)

The recoveries for the spiked compounds must be within statistical (or otherwise specified) limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze the LCS to determine if instrumental conditions or analytical preparation was the cause. If the LCS is still non-compliant notify the project manager using an NCM, to determine if re-extraction is required of all associated samples. Unless otherwise directed, samples will not be extracted outside the hold time and the data will be submitted with the appropriate narration. Note: State specific or program specific acceptance criteria may be required, check the analyst backlogs and/or the Project/Job notes in LIMs to determine if any custom limits are applicable.

8.2.4 LCS Duplicates (LCDs)

The LCD must meet all control limits as LCS in addition to limits set for precision (same corrective action as the LCS.

8.2.5 Method Blank (MB)

The MB should not contain any target compounds at or above the reporting limit. If non-compliant, then the MB should be reanalyzed to determine if instrument contamination was the cause. If the MB is still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCM and re-analyze all associated samples containing target compounds at <10x amount found in the MB. If a sample contains target compounds at ≥ 10x amount found in the MB, or if target compounds are not detected in the sample, then that sample does not require re-extraction and the results may be reported without qualification. If the samples are outside the extraction holding time, then contact the project manager using an NCM. Unless directed otherwise, samples will not be extracted outside the holding time and the data will be submitted with appropriate narration.

8.2.6 Matrix Spike (MS)

The recoveries for the spiked compounds should be within statistical limits. If non-compliant, check the calculations and spike preparation for documentable errors. If no errors are found, and the associated LCS is within advisory limits, then sample matrix effects are the most likely the cause.

8.2.7 Matrix Spike Duplicate (MSD)

See MS for recovery, RPDs should be within laboratory control limits. If non-compliant, check calculations for documentable errors. Check unspiked sample results and surrogate recoveries for indications of matrix effects. If significant differences exist between the MS and MSD, reanalysis of the sample spikes may be necessary.

8.2.8 Surrogate Spike

Both surrogates should be within laboratory control limits. If non-compliant, check the calculations and spike preparation for documentable errors. If no errors are found, and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely the cause. Any samples with surrogate recoveries outside the control limits with no visible chromatographic cause should be reanalyzed to determine if an injection error was the cause for the out of control recovery. If the surrogate recoveries in the associated MS and LCS are not within limits, and the samples are within the extraction holding time, then re-extract and reanalyze all associated samples. If the samples are outside the extraction holding time, then contact the project manager using and NCM. Unless otherwise directed, samples will not be re-extracted out of hold time and data will be submitted with appropriate narration.

8.2.9 Initial / Continuing Calibration Verification (ICV / CCV)

The response factors of the initial calibration check standard should not vary from the response factors of the corresponding standard from the calibration curve by more than ± 15% on at least one column for each target compound, and on both columns for any compound reported positive. If any of the target compounds fail this criterion, then the standard is considered to be non-compliant. If the standard is non-compliant, then reanalyze the standard. If the standard is still non-compliant, change inlet liner and o-ring, remove 1-2 inches from column (or guard column if present) and condition the column for 1-2 hours. If the standard is still non-compliant, then prepare a new standard. If the new standard is non-compliant, then recalibrate the instrument with a new curve. All target compounds which fail this criterion should be narrated as showing increased sensitivity, and how this might bias the data. Notify the project manager of any non-compliance using an NCM. Unless directed otherwise, samples will not be analyzed outside of holding time and the data will be submitted with appropriate narration.

8.2.10 Evaluation Mix B (EVAL B) PEM

If the degradation of 4,4'-DDT or Endrin is ≥ 15%, corrective actions must be taken. If the problem is not resolved by these actions, perhaps a new standard is necessary. Finally, a new column may be the solution to the problem.

Through out the analytical sequence, Evaluation Mix B must be run at intervals defined in Table 3. If the breakdown of Endrin or 4,4'-DDT is ≥ 15%, corrective actions must be taken and all samples run after the breakdown was unacceptable and before it was back in control, must be re-analyzed.

8.2.11 Retention Time (RT) Windows

Evaluate the data for usability based on a comparison with the standards run during the analytical sequence. Also, consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question.

When resolution between peaks is not ≥25%, adjust the carrier gas flow and/or column temperature to optimize conditions so this criterion may be met. This is usually a trial and error method to find the right combinations. Conditions should not vary much from what is described in Section 7.5.1. If they do, notify your section manager or supervisor because there could be a serious instrument problem.

If a continuing standard has any analytes that are outside of their daily windows, corrective actions must be taken before continuing. Check the septa first and make sure it is tight before any other possibilities are investigated. Check the press connectors, discoloration of the guard column (if present) or check the carrier gas and the connections to the instrument for possible leaks.

IF AT ANY TIME THE ANALYST IS UNCERTAIN AS TO WHAT ACTIONS TO TAKE OR HOW TO PERFORM SUGGESTED MAINTENANCE, PLEASE SEE YOUR SECTION MANAGER OR SUPERVISOR. IF SOMETHING UNUSUAL IS HAPPENING THAT IS NOT DESCRIBED IN THIS SECTION, SEE YOUR SECTION MANAGER OR SUPERVISOR. COSTLY (AS IN MONEY AND TIME) DAMAGE CAN RESULT TO THE INSTRUMENT, DETECTOR, OR COLUMN IF MAINTENANCE IS NOT PERFORMED CORRECTLY.

9.0 DATA ANALYSIS AND CALCULATIONS

The concentration of each analyte in the sample may be determined using the following calculation:

9.1 Concentration (ppb) $= \frac{(A_1)(V_t)(Df)]}{[(V_i)(V_s)(D)][CF]}$

Where:
A_1 = response for the analyte in the sample (area or height)
V_i = volume of extract injected (mL).
Df = dilution factor (if applicable)
V_t = volume of total extract (mL).
V_s = volume/weight of sample extracted
D = (100 - % Moisture) / 100
CF = Average calibration factor from the initial calibration curve

Toxaphene and Technical Chlordane are all multi-response compounds. Peak shape and pattern are evaluated more so than just retention times when calculating the concentrations of these analytes.

NOTE: All dry weight corrections are made in LIMs at the time the result is calculated in the analytical batch.

9.2 %RSD $= \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$

Where,
Standard Deviation $= \left[\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}\right]^{1/2}$

Where,
x_1 = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

9.3 % Difference $= \frac{CF_{ccv} - \text{Mean CF}}{\text{Mean CF}} \times 100$

Where:
CF_{ccv} = CF of the CCV
Mean CF = Mean CF from the ICAL.

9.4 Surrogate Percent Recovery $= \frac{Q_d}{Q_a} \times 100$

Where:
Q_d = Quantity determined by analysis
Q_a = Quantity added to sample/blank

9.5 Spike Recovery $= \frac{SR}{SA} \times 100$

Where:
SR = Spike result
SA = Spike added

9.6 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.7 Precision: $\%D = \frac{|B_1 - B_2|}{\text{Mean of } B_1 \,\&\, B_2} \times 100$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCS)

9.8 Endrin / DDT Breakdown

$$\text{\% breakdown of 4,4'-DDT} = \frac{\text{total DDT degradation peak area/height (DDE + DDD)}}{\text{total DDT peak area/height (DDT + DDE + DDD)}} \times 100$$

$$\text{\%breakdown for Endrin} = \frac{\text{total Endrin degradation peak area/height (Endrin aldehyde + Endrin ketone)}}{\text{total Endrin peak area/height (Endrin + Endrin aldehyde + Endrin ketone)}} \times 100$$

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Flammable Vials" waste stream.
- All samples containing >50 ppm of PCB material will be segregated into the container marked "PCB Waste".
- Extreme care should be taken not to mix waste streams. Putting Pest/PCB waste into non-PCB waste streams is a serious compliance violation and non-PCB waste into PCB waste streams adds excessive cost to waste disposal budget.
- Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Table 1. Target Compounds and Reporting Limits
Table 2. ICAL Form 6 - Standard Concentrations
Table 3. Example: Analytical Sequence

Attachment 1. TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points'
Attachment 2. Example: Worklist Run Log Report / Maintenance Log
Attachment 3. Example: Data Review Checklist
Attachment 4. TestAmerica Corporate SOP, CA-Q-S-002, Acceptable Manual Integration Practices and example documentation.
Attachment 5: TestAmerica Corporate Procedure, CA-T-P-003, Reporting Results for Methods that Require Second-Column Confirmation.

14.0 REVISION HISTORY

- Revision 16 updated on 09/20/13
- Remove reference to the DoD QSM criteria
- Update standard vendor and concentration preparation information.
- Section 8.2.2 updated with 8081A vs. 8081B ICV criteria.
- Section 9.8 updated to include peak area or peak height allowance.

Table 1.

Example: Target Compounds and Reporting Limits (021-001 to 021-002)

MDL W_LVI	Organochlorine Pesticides (GC)	8081B

Analyte Description	CAS Number	RL	LOD	MDL	Units	LCS LCL	LCS UCL	RPD	MS LCL	MS UCL	RPD	Surr LCL	Surr. UCL	Units
alpha-BHC	319-84-6	0.04	0.02	0.0026	ug/L	72	113	20	72	113	20			%
beta-BHC	319-85-7	0.04	0.02	0.0102	ug/L	77	115	20	77	115	20			%
delta-BHC	319-86-8	0.04	0.02	0.0103	ug/L	77	122	20	77	122	20			%
gamma-BHC (Lindane)	58-89-9	0.04	0.02	0.0056	ug/L	72	118	20	72	118	20			%
Heptachlor	76-44-8	0.04	0.02	0.0135	ug/L	60	110	20	60	110	20			%
Aldrin	309-00-2	0.04	0.02	0.0053	ug/L	58	110	20	58	110	20			%
Heptachlor epoxide	1024-57-3	0.04	0.02	0.0138	ug/L	80	119	20	80	119	20			%
Endosulfan I	959-98-8	0.04	0.02	0.0041	ug/L	59	113	20	59	113	20			%
Dieldrin	60-57-1	0.04	0.02	0.0129	ug/L	75	119	20	75	119	20			%
4,4'-DDE	72-55-9	0.04	0.02	0.0038	ug/L	74	120	20	74	120	20			%
Endrin	72-20-8	0.04	0.02	0.0142	ug/L	73	121	20	73	121	20			%
Endosulfan II	33213-65-9	0.04	0.02	0.0028	ug/L	68	120	20	68	120	20			%
4,4'-DDD	72-54-8	0.04	0.02	0.0133	ug/L	78	129	20	78	129	20			%
Endosulfan sulfate	1031-07-8	0.04	0.02	0.0117	ug/L	75	136	20	75	136	20			%
4,4'-DDT	50-29-3	0.04	0.02	0.0032	ug/L	61	122	20	61	122	20			%
Methoxychlor	72-43-5	0.08	0.04	0.023	ug/L	64	126	20	64	126	20			%
alpha-Chlordane	5103-71-9	0.04	0.02	0.0044	ug/L	75	114	20	75	114	20			%
gamma-Chlordane	5103-74-2	0.04	0.02	0.0072	ug/L	74	116	20	74	116	20			%
Toxaphene	8001-35-2	0.4	0.2	0.2	ug/L									%
Endrin aldehyde	7421-93-4	0.04	0.02	0.0082	ug/L	76	129	20	76	129	20			%
Endrin ketone	53494-70-5	0.04	0.02	0.017	ug/L	75	135	20	75	135	20			%
DCB Decachlorobiphenyl	2051-24-3				ug/L							30	131	%
Tetrachloro-m-xylene	877-09-8				ug/L							44	120	%
Chlordane (technical)	57-74-9	0.08	0.04	0.04	ug/L									%

MDL W_LVI	Liquid-Liquid Extraction (Separatory Funnel)	3510C_LVI

(021-001)

MDL S GC Extracatables	Organochlorine Pesticides (GC)	8081B

Analyte Description	CAS Number	RL	LOD	MDL	Units	LCS LCL	LCS UCL	RPD	MS LCL	MS UCL	RPD	Surr LCL	Surr. UCL	Units
alpha-BHC	319-84-6	1.7	0.83	0.424	ug/Kg	53	110	30	53	110	30			%
beta-BHC	319-85-7	1.7	0.83	0.518	ug/Kg	65	110	30	65	110	30			%
delta-BHC	319-86-8	1.7	0.83	0.526	ug/Kg	50	110	30	50	110	30			%
gamma-BHC (Lindane)	58-89-9	1.7	0.83	0.362	ug/Kg	55	110	30	55	110	30			%
Heptachlor	76-44-8	1.7	0.83	0.701	ug/Kg	50	110	30	50	110	30			%
Aldrin	309-00-2	1.7	0.83	0.693	ug/Kg	54	110	30	54	110	30			%
Heptachlor epoxide	1024-57-3	1.7	0.83	0.594	ug/Kg	50	122	30	50	122	30			%
Endosulfan I	959-98-8	1.7	0.83	0.731	ug/Kg	51	110	30	51	110	30			%
Dieldrin	60-57-1	1.7	0.83	0.229	ug/Kg	63	110	30	63	110	30			%
4,4'-DDE	72-55-9	1.7	0.83	0.277	ug/Kg	64	110	30	64	110	30			%
Endrin	72-20-8	1.7	0.83	0.231	ug/Kg	59	110	30	59	110	30			%
Endosulfan II	33213-65-9	1.7	0.83	0.271	ug/Kg	56	110	30	56	110	30			%
4,4'-DDD	72-54-8	1.7	0.83	0.333	ug/Kg	66	110	30	66	110	30			%
Endosulfan sulfate	1031-07-8	1.7	0.83	0.305	ug/Kg	63	120	30	63	120	30			%
4,4'-DDT	50-29-3	1.7	0.88	0.88	ug/Kg	50	115	30	50	115	30			%
Methoxychlor	72-43-5	8.3	4.2	0.324	ug/Kg	52	119	30	52	119	30			%
alpha-Chlordane	5103-71-9	1.7	0.845	0.845	ug/Kg	61	110	30	61	110	30			%
gamma-Chlordane	5103-74-2	1.7	0.83	0.438	ug/Kg	62	110	30	62	110	30			%
Endrin aldehyde	7421-93-4	1.7	0.83	0.281	ug/Kg	56	110	30	56	110	30			%
Endrin ketone	53494-70-5	1.7	0.83	0.378	ug/Kg	59	120	30	59	120	30			%
DCB Decachlorobiphenyl	2051-24-3				ug/Kg							56	128	%
Tetrachloro-m-xylene	877-09-8				ug/Kg							45	112	%
Toxaphene	8001-35-2	16.7	8.3	7.04	ug/Kg									%
Chlordane (technical)	57-74-9	6.7	3.3	3.25	ug/Kg									%

MDL S GC Extracatables	Automated Soxhlet Extraction	3541

(021-002)

Table 2.

Example: ICAL Form 6 - Standard Concentrations (022-001 to 022-002)

TestAmerica Laboratories
Initial Calibration Report

Method: \\Chi-svr07\ChromData\GC15-16\20130920-19098.b\inst 15_16.m
Instrument: INST15-16 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: Genie Last Modified: 24-Sep-2013 07:09:20
No.Compounds:38

Initial Calibration Batches

Ical Batch: \\Chi-svr07\ChromData\GC15-16\20130816-18467.b
Inj Date : 17-Aug-2013 12:56:40, Sublist: chrom-inst 15_16*sub3

Ical Batch: \\Chi-svr07\ChromData\GC15-16\20130823-18617.b
Inj Date : 23-Aug-2013 14:10:10, Sublist: chrom-inst 15_16*sub6

Ical Batch: \\Chi-svr07\ChromData\GC15-16\20130909-18897.b
Inj Date : 09-Sep-2013 14:23:38, Sublist: chrom-inst 15_16*sub1

Limit Group: GC_Pest_8081B

Detector 1: Ch-A

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
$ 30 Tetrachloro-m-xylene	336241138	322111210	295004000	277048266	269668730	268030252		294683933		Avg 9.7
7 alpha-BHC	492228400	489109500	489860650	500081550	507475363	525786888		500757058		Avg 2.8
29 Simazine	2295412	2239639	2196956	2167752	2189303			2217813		Avg 2.3
21 gamma-BHC (Lindane)	481062600	471795800	469158350	470989125	473048800	486636988		475448610		Avg 1.4
9 Atrazine	2191869	2230990	2204013	2153210	2138147			2183646		Avg 1.7
10 beta-BHC	230831600	215063300	201670150	190323025	182861363	181646519		200399326		Avg 9.7
13 delta-BHC	467302200	462218500	466262450	470931375	475909688	489872031		472082707		Avg 2.1
11 Chlordane (technical-1	14013100	15414680	14507320	15110460	15191730			14847458		Avg 3.9
11 Chlordane (technical-2	20054900	18001380	16390840	17659877	17229734			17867346		Avg 7.6
11 Chlordane (technical-3	48359100	50676100	46721010	49254647	48682690			48738709		Avg 2.9
11 Chlordane (technical-4	69733200	77075320	71436540	75436367	74339750			73604235		Avg 4.1
11 Chlordane (technical-5	16789900	14716120	13426700	14400340	14221730			14710958		Avg 8.5
23 Heptachlor	442014800	430449900	420988700	417358775	415458638	418909425		424196706		Avg 2.4
6 Aldrin	427178000	419807000	414274400	413416425	412939600	420060569		417945999		Avg 1.3
5 Alachlor	49581980	49195500	48186200	45679635	44440250			47416713		Avg 4.8
25 Isodrin	288803400	297100286	298805400	301337450	303757643			297960836		Avg 1.9
24 Heptachlor epoxide	413521600	403122500	392525350	384577575	376550850	373676625		390662417		Avg 4.0
22 gamma-Chlordane	391978800	385697500	379019900	377110825	377561938	386118175		382914523		Avg 1.6
8 alpha-Chlordane	387373000	379444000	370233700	365421400	363430875	369414969		372552991		Avg 2.4
3 4,4'-DDE	474262600	522604500	471343750	488900150	530461113	487039638		495768625		Avg 5.0
15 Endosulfan I	306419000	251169200	299058000	279951725	230016875	275669019		273713970		Avg 11
14 Dieldrin	385223400	383466400	380170150	380244800	377394763	378970044		380911593		Avg 0.76
18 Endrin	53074200	51713300	52022000	51920000	52237988	50507550		51912506		Avg 1.6
26 Kepone	162247860	115060486	124927480	157792838	174100211			146825775		Avg 17
2 4,4'-DDD	299737200	302029900	300562000	301959775	303063363	309161950		302752365		Avg 1.1
16 Endosulfan II	359281200	349180000	336685200	331280450	325009338	323166394		337433764		Avg 4.2
31 Toxaphene-1	14478500	14628746	14891104	15515967	15142559			14931375		Avg 2.8
31 Toxaphene-2	15602740	15964892	17273869	17532632	17817797			16838386		Avg 5.9
31 Toxaphene-3	16321820	16567374	17275399	18198199	17676993			17207957		Avg 4.5
31 Toxaphene-4	8305280	9270940	9680818	10399959	10240031			9579406		Avg 8.8
31 Toxaphene-5	10020440	11186018	11877876	12570967	12356850			11602430		Avg 8.9
4 4,4'-DDT	262603000	270575500	276323800	282833800	289913063	300755125		280500715		Avg 4.9
19 Endrin aldehyde	313753400	302769200	286702650	281699175	274962013	270299913		288364392		Avg 5.8
27 Methoxychlor	173689000	162152100	155756500	149713000	145780025	143485325		155095992		Avg 7.3
17 Endosulfan sulfate	357206800	343385500	326075700	320201900	314592250	313221963		329114019		Avg 5.3
20 Endrin ketone	386580800	382180800	372269550	370068375	363625975	361206850		372655392		Avg 2.7
$ 12 DCB Decachlorobiphenyl	354500600	330589700	311875400	297863800	290421838	291698250		312824931		Avg 8.1

(022-001)

Detector 2: Ch-B

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
$ 30 Tetrachloro-m-xylene	425901669	410131488	386762091	375260927	367271471	370731959		389343267		Avg 6.1
7 alpha-BHC	639326200	636871700	633084400	643070125	656314075	714846688		653918865		Avg 4.7
29 Simazine	1774083	1748146	1719584	1685636	1727155			1730921		Avg 1.9
21 gamma-BHC (Lindane)	572145000	570195700	569831500	577400800	587738038	645434831		587124311		Avg 5.0
9 Atrazine	1800744	1768578	1710145	1680726	1682062			1728451		Avg 3.1
10 beta-BHC	259457000	242887700	229601050	222062925	217406725	220973550		232064825		Avg 7.0
13 delta-BHC	799474200	798290900	799743650	812965300	837416525			809578115		Avg 2.1
11 Chlordane (technical-1	26767600	21962840	19870580	19401760	19465146			21493585		Avg 15
11 Chlordane (technical-2	31932700	26838820	24409620	24229357	24001876			26282475		Avg 13
11 Chlordane (technical-3	57443000	60271800	55152330	56216083	56740944			57164831		Avg 3.4
11 Chlordane (technical-4	40946200	44022520	40226420	40753230	41058768			41401428		Avg 3.6
11 Chlordane (technical-5	44958700	48423300	44189180	45403537	45928126			45780569		Avg 3.5
23 Heptachlor	396286400	408135600	393577550	396317475	405874900	Disabled		400038385		Avg 1.6
6 Aldrin	517576000	514063500	505857850	507225700	508433913	533859481		514502741		Avg 2.0
5 Alachlor	47522200	45500143	43259150	39773130	38199557			42850836		Avg 9.0
25 Isodrin	370664000	369954143	368331500	367824500	370222143			369399257		Avg 0.34
24 Heptachlor epoxide	482036800	473226600	452092500	445847425	436278575	436230744		454285441		Avg 4.2
22 gamma-Chlordane	470272800	466988300	457730200	460063225	466570713	520372431		473666278		Avg 4.9
8 alpha-Chlordane	452583800	450805900	439888900	440530300	446899275	457889669		448099641		Avg 1.6
3 4,4'-DDE	432066000	437375400	433333000	438215075	448746013	489107756		446473874		Avg 4.9
15 Endosulfan I	411640600	401382800	386596750	380682050	370592463	400665731		391926732		Avg 3.9
14 Dieldrin	450833400	452736000	441602500	440733225	435880663	450276169		445343659		Avg 1.5
18 Endrin	84749800	86360400	85779550	85913550	84542975	86198825		85590850		Avg 0.89
26 Kepone	129954430	86067779	105482685	150524183	185973034			131600422		Avg *30 R1
2 4,4'-DDD	365819000	368358200	362357450	365612575	369410000	391268919		370471024		Avg 2.8
16 Endosulfan II	401405600	394050600	376469000	372124675	363897588	372766938		380119067		Avg 3.8
31 Toxaphene-1	15347760	14860254	15220506	15691419	15582665			15340521		Avg 2.1
31 Toxaphene-2	22085080	21050356	21904511	23069012	23037295			22229251		Avg 3.8
31 Toxaphene-3	19836260	19963698	21037490	22459098	22628155			21184940		Avg 6.3
31 Toxaphene-4	22188280	22956940	24402478	26055048	25987182			24317986		Avg 7.2
31 Toxaphene-5	9447700	9478728	9991016	10620909	10589355			10025542		Avg 5.7
4 4,4'-DDT	234801600	249836400	251402900	265060525	277107888	303246956		263576045		Avg 9.2
19 Endrin aldehyde	344709000	336465100	315485400	312824350	302621125	301318863		318903973		Avg 5.6
27 Methoxychlor	137337000	140662700	134232300	132760925	131151363	133060944		134867539		Avg 2.6
17 Endosulfan sulfate	396262000	391268800	374421950	373445025	370620988	382898156		381486153		Avg 2.7
20 Endrin ketone	392421200	390454000	377394050	377069825	371324013	377541094		381034030		Avg 2.2
$ 12 DCB Decachlorobiphenyl	352371800	340156800	319315750	313031025	311365313	335479575		328620044		Avg 5.0

ICalib Error Legend
R1, Curve Coefs Fail the Rule Error Limit Test

(022-002)

Table 3.

Example: Analytical Sequence

Quantitation & Confirmation	
1. EVALB (PEM)	28. 20 Samples or 12 hours*
2. STD6 AB	29. CCV AB
3. STD5 AB	30. CCV TOXAPH
4. STD4 AB	31. CCV Chlordane
5. STD3 AB	32. EVALB (PEM)
6. STD2 AB	33. 20 Samples or 12 hours*
7. STD1 AB	34. CCV AB
8. ICV AB	35. CCV TOXAPH
9. STD5 TOXAPH	36. CCV Chlordane
10. STD4 TOXAPH	37. EVALB (PEM)
11. STD3 TOXAPH	
12. STD2 TOXAPH	
13. STD1 TOXAPH	
14. STD5 Chlordane	
15. STD4 Chlordane	
16. STD3 Chlordane	
17. STD2 Chlordane	
18. STD1 Chlordane	
19. CCV AB	
20. CCV TOXAPH	
21. CCV Chlordane	
22. EVALB (PEM)	
23. 20 samples or 12 hours*	
24. CCV AB	
25. CCV TOXAPH	
26. CCV Chlordane	
27. EVALB (PEM)	

*may inject up to 20 samples or 12 hours from the start of the calibration verification standards

NOTE: MB must always be run on the confirmation column.

NOTE: Isodrin & Kepone linearities (or any "non-routine" compound requested by a client) must be run at the beginning of the analytical sequence and must be alternated throughout the sequence as a CCV.

Attachment 1.

TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (024-001 to 024-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

Technical Services Director	06/18/2012 Date	Quality and EH&S Director	12/21/2012 Date

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1.0 PURPOSE

This policy describes TestAmerica Laboratories, Inc. requirements for the determination of the number of points and removal of points from calibration curves.

2.0 SCOPE

This policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP), then that requirement will be used for the defined procedure.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the DQOs for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a single point at the reporting level.

Examples:

A. Required to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by HPLC-UV at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the DQOs will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in the laboratory for meeting a regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A 5-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where the technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS's used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the QA Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of points from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24-hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internal standards show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (All analytes in spike mix must show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection.)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers), the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.
- Revision 2.1, dated 11 June 2012.
  - General clarifications – no procedural changes.

Attachment 2.

Example: Worklist Run Log Report / Maintenance Log (025-001 to 025-002)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 092013 Worklist Num: 19098
Instrument: INST15-16 Method: inst 15_16
Batch Directory: \\Chi-svr07\ChromData\GC15-16\20130920-19098.b
Anaylysis Type: SemiVOA Creator: Gibson, Patti J
Inj Volume: 1.00 Inj Vol Units: ul

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
PEM	500-0019098-001	PEM		20-Sep-2013 09:12:47	091813_036.D		1.0		sv
CCV TOX	500-0019098-002	CCV		20-Sep-2013 09:32:22	091813_037.D		1.0		sv
CCV AB	500-0019098-003	CCV		20-Sep-2013 09:52:11	091813_038.D		1.0		sv
CCV CHLOR	500-0019098-004	CCV		20-Sep-2013 10:11:47	091813_039.D		1.0		sv
MB 500-203461/1-A	500-0019098-005	MB		20-Sep-2013 10:31:21	091813_040.D		1.0		sv
LCS 500-203461/2-A	500-0019098-006	LCS		20-Sep-2013 10:50:55	091813_041.D		1.0		sv
LCS 500-203461/3-A	500-0019098-007	LCS		20-Sep-2013 11:10:29	091813_042.D		1.0		sv
LB 500-203192/1-E	500-0019098-008	LB		20-Sep-2013 11:30:04	091813_043.D		1.0		sv
LB3 500-203410/1-C	500-0019098-009	LB3		20-Sep-2013 11:49:37	091813_044.D		1.0		sv
500-63126-A-1-F	500-0019098-010	Client		20-Sep-2013 12:09:27	091813_045.D		1.0	JL-JLB041-ST01-09	sv
CCV TOX	500-0019098-011	CCV		20-Sep-2013 12:29:00	091813_046.D		1.0		sv
CCV AB	500-0019098-012	CCV		20-Sep-2013 12:48:34	091813_047.D		1.0		sv
CCV CHLOR	500-0019098-013	CCV		20-Sep-2013 13:08:09	091813_048.D		1.0		sv
PEM	500-0019098-014	PEM		20-Sep-2013 13:27:44	091813_049.D		1.0		sv
500-62771-B-1-I	500-0019098-015	Client		20-Sep-2013 13:47:18	091813_050.D		1.0	WP-1 (3')	sv
500-62771-B-1-J MS	500-0019098-016	MS		20-Sep-2013 14:06:52	091813_051.D		1.0		sv
500-62771-B-1-K MS	500-0019098-017	MS		20-Sep-2013 14:26:39	091813_052.D		1.0		sv
500-62771-B-2-J	500-0019098-018	Client		20-Sep-2013 14:46:13	091813_053.D		1.0	WP-2 (4')	sv
500-62771-B-3-K	500-0019098-019	Client		20-Sep-2013 15:05:46	091813_054.D		1.0	WP-3 (5')	sv
500-62872-A-1-G	500-0019098-020	Client		20-Sep-2013 15:25:21	091813_055.D		1.0	Wash 200+55 RT	sv
500-62872-A-2-H	500-0019098-021	Client		20-Sep-2013 15:44:55	091813_056.D		1.0	CLK 24+27 RT	sv
CCV TOX	500-0019098-022	CCV		20-Sep-2013 16:04:29	091813_057.D		1.0		sv
CCV AB	500-0019098-023	CCV		20-Sep-2013 16:24:01	091813_058.D		1.0		sv
CCV CHLOR	500-0019098-024	CCV		20-Sep-2013 16:43:48	091813_059.D		1.0		sv
PEM	500-0019098-025	PEM		20-Sep-2013 17:03:22	091813_060.D		1.0		sv

(025-001)

TestAmerica Chicago
GC Extractables Instrument Maintenance Log

Page No.:_____
Instrument No.: 41 / 42

Routine/Informal Maintenance includes: Glass Y-connectors checked for leaks; periodic system check for leaks, frayed wires and a general overview; flows checked

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Reviewer Signature:_____ Date:_____

Attachment 3.

Example: Data Review Checklist
(026-001 to 026-003)

TestAmerica
THE LEADERS IN ENVIRONMENTAL TESTING

Chicago
CHI-22-17-069/D-01/13

GC/HPLC Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
REPORTS:		
p-Flag report reviewed and accepted?		
Retention Time Study reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: <20% RSD; for 608 and 610 < 10% RSD		
Linear or Quadratic Regression curve fit for all >20% RSD R^2 >0.990		
Plots for all Linear Regressions reviewed?		
conc-intercept < RL; (<1/2 RL QAPP specific); <30% Error (Chrom: Details of Calibration per Analyte)		
Co-eluting peaks reviewed and accepted?		
All manual integrations reviewed found to be necessary, performed correctly and have documented reasons?		
Method:		
Method locked in Chrom?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct and locked in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 85-115% (15%D) criteria? 8015C; 8082A; 8081B (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date_____

Secondary Reviewer: _____ Date_____

(026-001)

TestAmerica Chicago

GC EXTRACTABLES / HPLC DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Analytical Method: a) GC _____ b) HPLC _____ Instrument (Primary / Confirmation):_____

Extraction Method: a) SW 3510 b) SW 3541 c) SW 3550 d) SW 3580 e) Other (_____)

Matrix: WATER / SOIL / SPLP-TCLP / Waste / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: _____ _____ _____ _____ _____
Import Batch Review: Is the Calibration ID Correct? Y N Report Status of Primary / Secondary correct? Y N CCV's are correct and properly linked to samples? Y N Calculation of all QC complete? Y N			
View All Data Review: *Samples:* Results reported for all samples? Y N Dilutions / Re-analyses are correctly represented? Y N NA Are flags correctly assigned? Y N *QA Summary: QC Association Summary* All required dilutions/re-analyses present? Y N All Samples linked to prep batch? Y N All Samples linked to batch QC? Y N *Lab QC:* All QC present and correctly represented? Y N *Surrogate*: All samples/QC present and correct? Y N			
Form 1 / View All Data: IF **original** and **re-analyses** are to be reported in LIMS: Suffix Used: Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Sample # Original Dilution Comments
FORM 2 / View All Data: Surrogate Recoveries within Limits? Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3 / View All Data: MS/MSD Recoveries Acceptable Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3 / View All Data: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

(026-002)

TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Criteria Met QAPP specific MB < ½ RL _____ NCM Ref Number: _____			
FORM 4: 1) Primary and Secondary Column info present? Yes No NA 2) All Samples Present Yes No NA 3) Sulfur and GPC clean-ups identified? Yes No NA 4) Header Information Correct? Yes No NA NCM Ref Number: _____			
FORM 8: Retention Time Window Criteria Met? Yes No NCM Ref Number: _____			
Form 10: 1) Retention Time Window Criteria Met? Yes No NA 2) All hits present/Concentrations Correct? Yes No NA 3) Are hits < 40%D between primary & secondary columns? Yes No (NCM Ref No.: _____)			
RAW DATA: 1) Raw Data Verified and Complete? Yes No NA			
2) Raw Data Matches Forms? Yes No NA			
3) Sample info complete-% solids, Smp.Wt, F.V.?			
4) Prep/Extraction Data info verified in Raw Data?			
Form 6: Initial Calibration Criteria met and Complete? Samples linked to ICAL? _____			
Form 7: CCV criteria met method criteria Samples linked to CCV? _____ NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Blank / LCS / LCSD Yes No 2) MS/MSD Yes No NA 3) MRL Check Required: Yes No Limits Applied: _____			
Manual Integration reports (before and afters) present and reasons correctly documented and approved?			
Prep / Sample Summary pages Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required / Properly Documented? Yes No			
Manual Calculation of On Column result: Peak Height or Area of Analyte Average Peak Height or Area from ICAL			Sample : _____ Compound: _____
JOB STATUS: 1) Primary Reviewer/Analyst: 1st / 2nd Level			
2) Secondary Reviewer/Supervisor: Lab Complete			
Additional Comments:			

Attachment 4.

TestAmerica Corporate SOP, CA-Q-S-002,
'Acceptable Manual Integration Practices'
(027-001 to 027-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

Raymond J. Frederici 08/01/2012
Raymond J. Frederici — Date
Quality and EH&S Director

Larry Penfold 08/01/2012
Larry Penfold — Date
Quality Compliance Director

Verl D. Preston 08/01/2012
Verl D. Preston — Date
Quality Assessment Director

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1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

(027-002)

4.5 **Baseline:** The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 **Peak:** An increase in signal from baseline to a maximum and then back to baseline.

4.7 **Coelution:** The concurrent elution of two or more compounds to the detector at the same time.

4.8 **Elution:** The process of movement of compounds from the chromatographic system.

4.9 **Carry-over:** Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 **Peak Tailing:** Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 **Interference Peaks or Fused Peaks:** Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important considerations in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters is necessary to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibrations standards, QC standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. This

process for before & after chromatograms is also required for OVAP-approved laboratories, unless instructed otherwise.

5.4.7.1 Re-integration technique marked, if available, on the data system.

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.

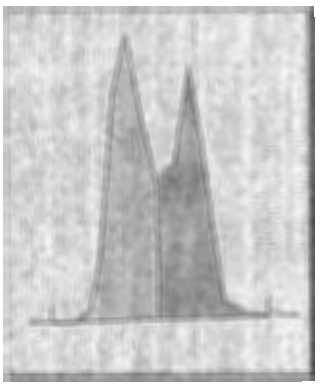
- Expanded section on data miner software to include Chrom Audit Miner
- Included reference to data miner training presentation.

- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples

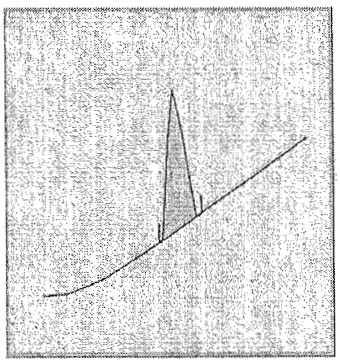
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

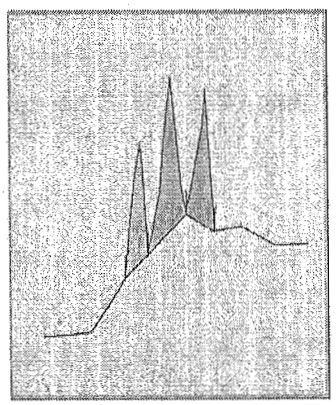
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.

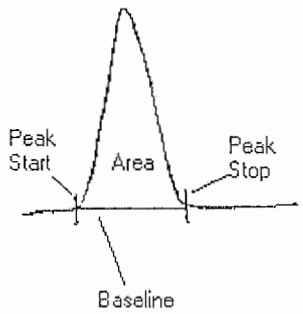
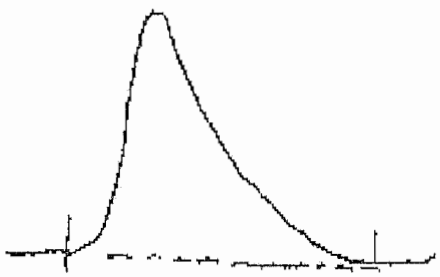


Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.

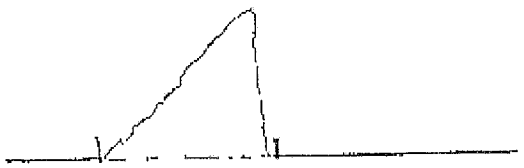
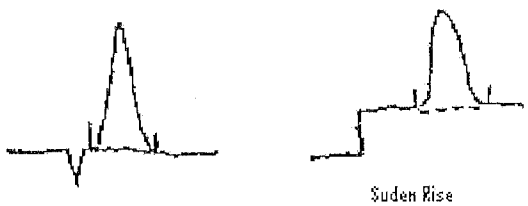


Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.

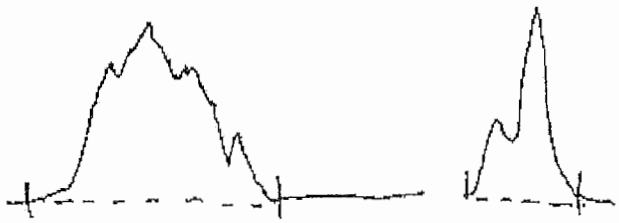
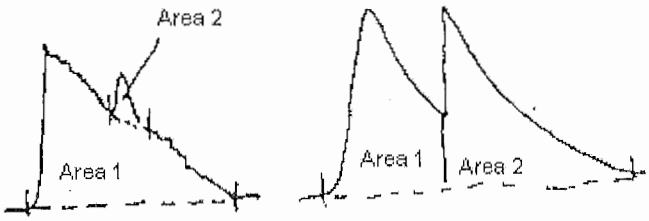


Ideal peak (Gaussian Shape) The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

(027-011)

Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	 Negative Dip Suden Rise

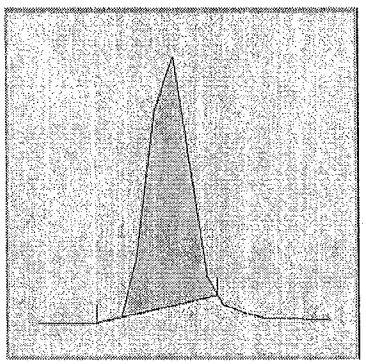
(027-012)

Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

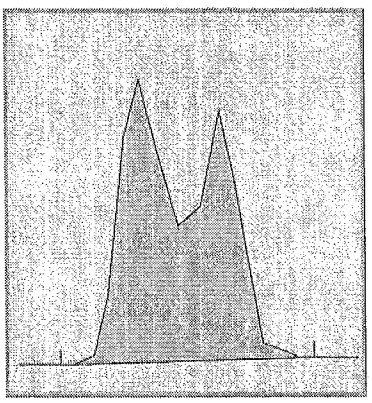
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

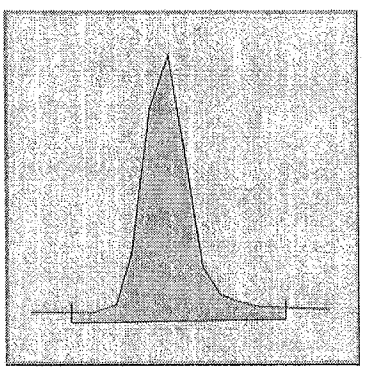
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.

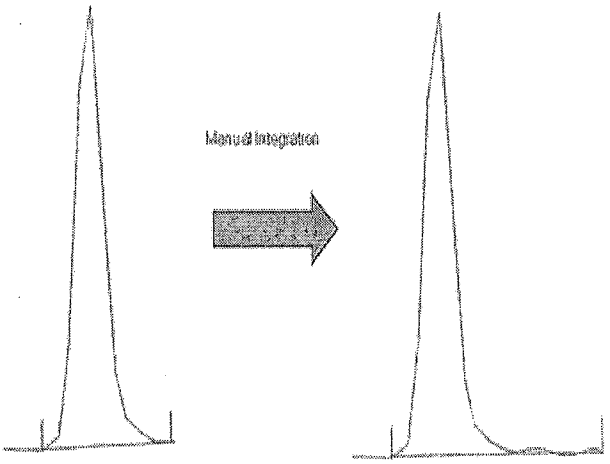


Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.

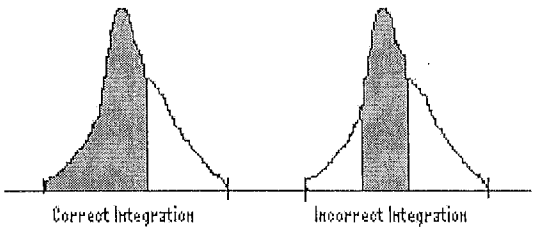


(027-014)

Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 5.

TestAmerica Corporate Procedure, CA-T-P-003, Reporting Results for Methods that Require Second-Column Confirmation (028-001 to 028-003)

Reporting Results for Methods that Require Second-Column Confirmation

Approvals (Signature/Date):

Technical Services Director	06/18/2012 Date	Quality and EH&S Director	12/21/2012 Date

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1.0 PURPOSE

This policy describes a standardized approach to reporting results for methods that require second-column confirmation, such as EPA SW-846 8000 series GC methods. This policy is designed to be in compliance with EPA methods 8000B and 8000C as well as EPA 600 series methods and is intended to be followed in the absence of QAPP or regulatory guidance to the contrary (e.g., DOD, DOE, and CLP).

2.0 SCOPE

This policy applies to all methods that require second-column (qualitative and quantitative) confirmations. This procedure is not applicable to qualitative only confirmations, such as Methods 8330 or 8330A, which report only from the primary column.

3.0 SAFETY

Refer to Laboratory-specific method SOPs.

4.0 DEFINITIONS

None.

5.0 PROCEDURE

5.1 The laboratory may designate a primary column for each analyte (in which case the second column is the confirmation column for each analyte), or may report the lower result from the two columns. The same column does not have to be primary for every analyte. Both columns are intended to be calibrated and meet calibration verification requirements.

5.2 Whichever approach is used, QC must be assessed and reported in the same way as the samples (i.e., report from the primary column or report the lower number).

5.3 40% RPD Criterion

5.3.1 When using the primary column option, if the RPD for the dual column results is $\leq$ 40%, then the result from the primary column is normally reported. The exception is situations where a chromatographic interferent is evident in the primary column chromatogram that is not present in the confirmation column chromatogram.

5.3.2 If using the primary column option and the RPD for the dual column results is > 40%, then the lower value is reported. (A RPD of >40% is clear evidence of chromatographic interference on the higher value). A case narrative comment or footnote discussing the RPD failure must be included in the final report.

5.4 Calibration Verification Criterion – Primary Column Option

5.4.1 If the CCV recovery for the primary column is within acceptance limits and the analyte is confirmed on the secondary column, but the secondary column is outside acceptance limits then results from the primary column may be reported with a case narrative comment or footnote.

5.4.2 If the CCV recovery for the confirmation column is within acceptance limits and the recovery for the primary column is in the range of +15% to +40%, then results may be reported from the confirmation column with a case narrative comment or footnote.

5.4.3 If the CCV recovery is below acceptance limits for the primary column, or the secondary column is below acceptance limits and the analyte is not confirmed, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.4.4 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.5 Calibration Verification Criterion – Lower Result Option

5.5.1 If the CCV recovery for the column with the lower result is within acceptance limits and the analyte is confirmed on the secondary column but the secondary column is outside acceptance limits, then results from the lower column may be reported with a case narrative or footnote.

5.5.2 If the CCV recovery for the higher result is within acceptance limits but the lower result column fails acceptance limits and the RPD is < 15%, then the higher result may be reported with a case narrative or footnote.

5.5.3 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving second-column confirmation are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 6 November 2007
  - Initial Release.
- Revision 1, dated 23 June 2009
  - No Changes
- Revision 2, dated 18 June 2012
  - Added quantitative column confirmation
  - Added limits for CCV failures on confirmation columns; and clarified language.

TITLE: Gas Chromatography - Semi-Volatiles Analysis of PCBs by SW-846 Method 8082 and 8082A

Approvals (Signature/Date):

Gene Orf Analyst, GC Chemist	Date: 9/24/13
Gary L. Rynkar Supervisor GC Extr./HPLC	Date: 9/24/13
Chris Hoham Env. Health & Safety Coor.	Date: 9/24/13
Terese A. Preston Quality Assurance Manager	Date: 9/24/13
Michael J. Healy Laboratory Director	Date: 9/24/13

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1.0 SCOPE / APPLICATION

This method is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in assorted matrices by SW-846 Methods 8000B/8082 and 8000B/8082A. Note: Method 8000C criteria may be required if designated in specific project QAPPs.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values ~2-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to MDL are special circumstances not to be confused with the previous statement. Attachment 1 provides a list of compounds analyzed by this method and their reporting limits.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

This method provides the gas chromatographic (GC) conditions and necessary standardization procedures for the detection of parts per billion (ppb) levels of Aroclors. The appropriate sample extraction must be performed prior to sample analysis (Refer to Section 7.3.1).

2.0 INTERFERENCES

2.1 Phthalates

Interference's by phthalate esters can pose a problem in PCB analysis. These compounds generally appear in the chromatogram as large late-eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Avoiding contact with any plastic materials can best minimize interferences from phthalates.

2.2 Co-extractions

Co-extracted interferences will vary considerably from sample to sample. Clean-ups are available to help eliminate or lessen interferences. All extracts analyzed for PCBs routinely receive a sulfuric acid cleanup. Additionally, florisil clean-ups, sulfur clean-ups, and GPC clean-ups may be performed (Refer to UP-SP-003 for complete details).

2.3 Glassware

All glassware/equipment used in the preparation of standards (volumetric flasks, pipettes, beakers, eppendorf tips) are rinsed well with the appropriate solvent and allowed to dry before use. After completion of standard preparation, unused amounts of standards are properly disposed of, and all glassware/equipment rinsed well with the appropriate solvent. Rinse/excess solvent is properly collected and disposed of. These procedures should eliminate positive interferences from these sources.

2.4 4,4-DDT (PEM)

Interference by the compound 4,4-DDT can pose a problem in the analysis of AR1254. The peak representing 4,4-DDT can elude at the same time as an AR1254 peak. A 4,4-DDT standard (PEM) is analyzed with each initial calibration in order that the AR1254 peaks chosen for quantitation are not interfered with by 4,4-DDT.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- PCBs are a bio-accumulating carcinogen. Extreme care should be taken when working with this material.
- Interior parts of GC's can be very hot. Care should be taken if adjusting instrument.
- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

3.2 Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

4.1 Gas Chromatographs

Gas Chromatographs (GC) are equipped with dual electron capture detectors (ECDs) (Ni_{63}). Each GC is equipped with an autosampler.

4.2 Column - Megabor

- Column 1/signal 0: RTX-5, 0.53 mm ID, 30 m length, maximum temperature 330°C, and 0.5 - 1.0 micron film thickness (or equivalent).
- Column 2/signal 1: RTX-CLPesticides2, 0.53 ID, 30 m length, maximum temperature of 330°C. Film thickness of 0.42 micron (or equivalent).
- Guard Column - Phenyl-methyl deactivated 5 meter, 0.53 mm I.D. (or equivalent).

Similar columns can be used if all QC criteria can be met.

Note: Signal 0 is designated as the primary column in TALs and signal 1 is designated as the secondary column in TALs. Additional guidance can be found in Attachment 6, the corporate SOP CA-T-P-003, *Reporting Results for Methods That Require Second-Column Confirmation.*

4.3 Data Acquisition / Processing

- Data Acquisition is achieved with Perkin Elmer NCI 900 in conjunction with TotalChrom for data acquisition.
- Processing is performed with Chrom.

5.0 REAGENTS AND STANDARDS

The preparation of all standards and QC solutions must be properly documented. A unique reagent code is created in the TALs reagent module. The information includes the name of the reagent, the concentration of the reagent, the date prepared, expiration date and the analyst who prepared the reagent. The reagent label used to label each reagent bottle contains the unique TALs code for that reagent bottle, its description, expiration date, prepped/received date, solvent used, prepared by and date created. The label also contains a bar code for scanning into the Chrom worklists, which then transfer to the TALs AD batches upon upload.

All standards and spikes must be stored at 4±2°C in glass bottles with Teflon-lined lids.

5.1 Solvents

Hexane, Acetone, Isooctane...Pesticide quality or better.

5.2 Intermediate Standards

All standards used are certified. The Aroclor 1016 and 1260 used for the initial calibration are verified against an alternate source [Second Source Verification (SSV)]. Aroclor standards are purchased as solutions at a level of ~1000 ug/mL. Appropriate dilutions are prepared such that the intermediate standards are at a level of 5.0 ug/mL in Hexane for all Aroclors 1016/1260. Aroclors 1016 and 1260 are added together into the same intermediate solution and is referred to as AR1660.

The surrogate compounds, Decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachloro-m-xylene (TCX), are purchased as a solution at a level of ~200 ug/mL. An intermediate standard is made at concentrations of the surrogates in the intermediate standard are at 2.0 ug/mL (1 mL to 100 mL FV) in Hexane. Intermediate standard solutions must be replaced 6 months from the date of preparation.

5.3 Calibration Standards

A standard containing a mixture of Aroclor 1016 and Aroclor 1260 (AR1660) will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 /1260 and surrogates DCB and TCX at a minimum of five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Working calibration standards are prepared in Hexane from the intermediate solutions. At least 5 levels of AR1660 are required, however, generally 6 levels are prepared. Concentrations range from 0.040 ng/uL to 1.00 ng/uL and surrogate concentrations range from 0.004 ng/uL to 0.08 ng/uL (Table 1). The five concentration levels are prepared through dilutions of the intermediate standards with hexane. One of the concentration levels should be at or below the established reporting limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

Other Aroclor standards (1221, 1232, 1242, 1248, 1254, 1262 and 1268) are purchased as separate solutions at a level of 1000 ug/mL. A single point for each is prepared through an intermediate standard at 5 ug/mL prepared by taking 10 mL to 100 mL final volume of the 1000 ug/mL standard. A subsequent dilution with hexane at the mid-point concentration level is made by using 0.5 mL of the intermediate standard solution brought up to 100 mL final volume.

Note: The laboratory may be required to perform a full 5-point calibration curve for Aroclors other than AR1016/AR1260. The analyst needs to monitor their backlog for special project/job notes that may be associated with their samples which may involve special project, state or program requirements.

5.4 Surrogates

The analyst must monitor the performance of the extraction, clean-up (when used), analytical system and the effectiveness of the method for each sample matrix by spiking each sample, matrix spike (MS), MS duplicate (MSD), laboratory control sample (LCS) and method blank (MB) with the surrogates. The surrogates used are Decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachloro-m-xylene (TCX).

6.0 CALIBRATION

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

Calibration pertains to both primary and secondary columns. Establish GC operating parameters equivalent to those indicated in Section 7.4.1. Prepare calibration standards using the procedures indicated in Section 5.3.

Calibration Controls	Sequence	Control Limit
Initial Calibration Standards	6 pt. curve of AR1660 prior to samples	≤20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source Verification (SSV) [1]	After initial calibration	± 15% Difference (8082) ± 20 % Difference (8082A)
Cont. Calib. Verif. (CCV) [2,3]	Beginning sample sequence, Every 10 samples*	± 15% Difference (8082) ± 20% Difference (8082A)
Retention Time Windows	Established with the initial calibration,	3 X Std. Deviation

[1] The SSV concentration corresponds to Level 3 of the linearity standards (Table 1).
[2] The CCV concentration corresponds to the Level 4 of the linearity standards (Table 1).
[3] The CCV is required at least once per 20 samples; however, it is strongly recommended that it be analyzed after every 10 samples, to reduce the number of reanalyses if the CCV is out-of-control.

6.1 External Standard Calibration

6.1.1 Prepare calibration standards as described in Section 5.3. Because of the sensitivity of the ECD, the injection port and column should always be cleaned prior to performing the initial calibration. When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts.

6.1.1.1 As noted in Section 5.3, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other 5 Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial 5-point calibration is performed using the mixture of Aroclors 1016 and 1260 described in Section 5.3.

6.1.1.2 Standards of the other five Aroclors are necessary for the pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the AR1660 mixture has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the 5 AR1660 standards.

A PEM standard contains 4,4-DDT at the 0.02 ng/uL level is analyzed to determine potential pattern interference with AR1254.

6.1.1.3 Specific client/project/state agency/program protocol may include the requirement of full 5-point initial calibrations for one or all of the additional Aroclors. In these cases (example DOD QSM) the specific Aroclor will be quantitated from the 5-point curve versus a single-point.

6.1.2 Record the peak area (or height) for each characteristic Aroclor peak to be used for quantitation. A minimum of 3 peaks, preferably 5 peaks, must be chosen for each Aroclor. The peaks must be characteristic of the Aroclor in question. 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 3 to 5 peaks should include at least one peak that is unique to that Aroclor.

6.1.3 Calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the equation below:

$$\text{Calibration Factor (CF)} = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Five sets of calibration factors will be generated for the AR1660 mixture, each set consisting of the calibration factors for each of the 3-5 peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least 3 calibration factors, one for each selected peak.

The calibration curve type in Chrom must be HtAverage for all the peaks for the AR1016 and AR1260 standards in order for the calibration factors for all other Aroclors to be calculated from a single mid-point calibration standard.

6.2 Retention Time Windows

Absolute retention times are used for the identification of PCBs as Aroclors. However, in addition to retention times, peak patterns play a large roll in the identification of Aroclors.

Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

6.2.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make 3 injections of all standard mixtures throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

6.2.2 Record the retention time for 3 to 5 major peaks for both Aroclor 1016 and Aroclor 1260 to 3 decimal places. Calculate the mean and the standard deviation of the 3 absolute retention times.

6.2.3 If the standard deviation of the retention times for a target compound is 0.000 (no difference between the 3 retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

6.2.4 The width of the retention time window is defined as $\pm$3x the standard deviation of the absolute retention times. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

6.2.5 Establish the center of the retention time windows from the calibration verification at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Retention time windows can be updated every 12 hours. However, they are usually only updated at the onset of a continuing calibration sequence or after maintenance has been performed.

6.2.6 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory and available for review.

6.2.7 The laboratory monitors the retention time for both surrogates throughout the analytical sequence.

7.0 PROCEDURE

7.1 Quality Control Checks

The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 samples	< Reporting Limit <1/2 RL Project QAPP
LCS	1 in 20 samples	Stat. Control Limits (Attachment 1)
LCD [1]	1 in 20 samples	Stat. Control Limits (Attachment 1)
Matrix Spike (MS)	1 in 20 samples	Stat. Control Limits (Attachment 1)
MS Duplicate (MSD)	1 in 20 samples	Stat. Control Limits (Attachment 1)
Surrogate	every sample	At least 1 within statistical limits.

[1] LCD is required only when insufficient sample is available for the MS/MSD or as requested by the client/project.

Note: State specific or program specific acceptance criteria may be required, check the Project/Job notes in LIMs to determine if any custom limits are applicable. Ex. For the state of South Carolina, the LCS results must meet a 70 – 130% recovery window.

7.2 Sample Preservation and Storage

All samples should be collected in a glass container with a Teflon-lined lid. If the lid does not have a Teflon-liner, aluminum foil should be placed between the jar and lid (dull side facing sample). All samples should be refrigerated (4 $\pm$ 2°C) up until extraction; as well as all extracts up until analysis.

Matrix	Reference	Preservation	Holding Time: To Extract (VTS)	Holding Time: After Extr. to Analysis
Soil/Sediment	\	\	14 days	\
Water	SW-846	Cool, 4 $\pm$ 2°C	7 days	40 days
Waste/Oil	/	/	14 days	/

VTS = verified time of sampling

7.3 Sample Preparation

7.3.1 Extraction Procedure

The matrix determines which extraction procedure to follow. Waters are extracted following the separatory funnel method (SOP UP-SP-3510); soils are extracted by soxhlet (SOP UP-SP-3541); wipes are extracted by sonication (SOP UP-SP-3550); and wastes and oils are by dilution (SOP UP-SP-3580).

7.3.2 Clean-up Procedure

Various clean-up procedures are available if interferences are present. A sulfuric acid cleanup is always performed on all PCB extracts. A sulfur cleanup using elemental Mercury is used to eliminate sulfur interference in extracts as needed. A Florisil cleanup and a GPC cleanup may also be used (SOP UP-SP-003).

7.4 Calibration / Standardization

7.4.1 GC Conditions

Instrument 23/24

- Carrier Gas (Hydrogen) 3 - 10 mL/min
- Make up Gas (Nitrogen) > 80 mL/min
- Column Temp Program 170 - 325°C (based on elution profile)
- Ramp 25-50°C/min
- Injector Temp 240°C and/or Cool on Column
- Detector Temp 300°C

Instrument 31/32

- Carrier Gas (Hydrogen) 3 - 10 mL/min
- Make up Gas (Nitrogen) > 80 mL/min
- Column Temp Program 170 - 325°C (based on elution profile)
- Ramp 25-50°C/min
- Injector Temp 240°C and/or Cool on Column
- Detector Temp 300°C

Instrument 47/48

- Carrier Gas (Hydrogen) 3 - 10 mL/min
- Make up Gas (Nitrogen) >70 mL/min
- Column Temp Program 170 - 325°C (based on elution profile)
- Ramp 25-50°C/min
- Injector Temp 240°C and/or Cool on Column
- Detector Temp 300°C

These conditions are subject to change based on instrument performance. The oven temperature and carrier gas flow may vary slightly between instruments and columns.

7.4.2 Verification of Calibration Curve

Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every 20 samples (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 the analysis sequence. For Aroclor analyses, the calibration verification standard should be a mixture of AR1660. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclor after the AR1660 mixture used for calibration verification throughout the analytical sequence.

Note: The analyst needs to review their backlog for project/job notes regarding special project or program requirements that may need to be applied to their samples.

7.4.2.1 The calibration factor for each analyte calculated from the calibration verification standard (CCV) must not exceed a difference of more than ±15% (8082) ± 20% (8082A) when compared to the mean calibration factor from the initial calibration curve.

$$\% \text{ Difference} = \frac{CF_{ccv} - \text{Mean CF}}{\text{Mean CF}} \times 100$$

Where:
CF_{ccv} = CF of the CCV
Mean CF = Mean CF from the ICAL.

7.4.2.2 If this criterion is exceeded for any calibration factor, inspect the GC to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

7.4.2.3 Calibration Evaluation and Acceptance Criteria

Refer to the TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (Attachment 2)

7.5 Qualitative identification

Qualitative identifications of target analytes are made by examination of the sample chromatograms versus the standard chromatograms from the initial calibration. If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area when overlapping peaks cause errors in area integration.

7.5.1 Each sample analysis must be bracketed with an acceptable initial calibration, verification standard(s) (each 12-hour shift), or calibration standards interspersed within the samples. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be re-injected.

7.5.2 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

7.5.3 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out-of-control; however, pattern recognition is critical in determining the presence of Aroclors in associated samples. Determine the cause of the retention time shift and correct the problem.

7.5.4 The identification of Aroclors using this method is based predominantly on peak patterns. In addition, to a lesser degree, agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards is utilized. Tentative identification of an Aroclor occurs when a specific pattern of peaks from a sample extract matches the specific pattern of peaks observed in a standard. Again, peak pattern plays a large part in determining the presence of Aroclors in samples. All tentative identifications are confirmed using a second GC column of dissimilar stationary phase if required, based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS. Peaks chosen for AR1254 are such that there is no interference from 4,4-DDT.

7.5.5 Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the peak patterns on both columns match an Aroclor pattern, then the target analyte identification has been confirmed.

7.6 Quantitation of PCBs as Aroclors

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

7.6.1 Use the individual Aroclor standards (not the AR1660 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

7.6.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen in Section 6.1 and the calibration model (linear or non-linear) established from the multi-point calibration of the AR1660 mixture. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

7.6.3 Weathering of PCBs in the environment and changes resulting from waste treatment processed may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. The quantitation of Aroclors may be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

7.6.4 Manual Integration Policy

In each case where manual integration has taken place, Chrom will apply the operator's name and date and reason for the manual integration to the electronic file. Manual integrations shall follow the TestAmerica Corporate SOP for Manual Integrations (CA-Q-S-002) located in Attachment 5. An example of one such integration and documentation is included in the attachment. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

Manual integrations are most often performed for the following reasons.

Peak Not Integrated	Split Peak
Sample Matrix Interference	Assign Peak
Column Bleed	Baseline Smoothing
Instrument Noise	Isomers
Peak Tail	Shouldering
Incomplete Integration	Other
Wrong Peak	

A peer review is performed on all manual integrations. A pdf of all the raw data including the before and after manual integrations are automatically imported into the TALs AD batch from Chrom. A summary of the MIs are generated in the TALs deliverable and is include in the data summary portion of the data package. The pdf of the MIs are automatically included in the Level 4 deliverable.

7.7 Preventive Maintenance

- The septa should be changed between each analyses sequence. The number of injections made plays a part in how frequently they should be changed. Generally, not more than 100-150 injections should be made without changing the septum.
- The entire system including Nitrogen and Hydrogen tanks should be periodically checked for leaks and frayed wires.
- Leak tests must be performed every six months on every ECD.

7.7.1 Suggested Maintenance

When any calibration, QC sample or analytical sample is outside the acceptance criteria, one or more of the following actions may be necessary.

- Inspect injection end of column for particulates and/or cracks.
- Condition the column - upper end of column temperature 285°C for 1 - 2 hours.
- Remove 1-6 inches from the injector and guard column. (if present)
- Make sure septum doesn't need to be changed.
- Change single-tapered liner (w/glass wool)

Refer to the instrument manuals if problems are encountered which can not be solved by routine maintenance. Record all maintenance in the maintenance logbook with return to control clearly documented.

7.8 Gas Chromatographic Analysis

7.8.1 Prior to initial calibration or continuing calibration if the instrument has been idle for a period of time, several primes shall be analyzed to prepare the inlet, column and 'Y' (if applicable) by removing possible active sites from these surfaces. The prime shall be the AR1660CCV4 level, this is equivalent to a concentration of 0.5 ug/mL each component AR1016 and AR1260. Priming the instrument at this level has been successful at removing active sites and higher concentrations of the primes do not appear to be necessary.

7.8.2 **Direct Injection** - For a capillary column, inject 1 - 2 uL of the sample extract using a solvent flush technique equipped with the autosampler.

7.8.3 If the response of a sample exceeds the linear range of the system dilute the extract and reanalyze. Over lapping peaks are not always evident when peaks are off scale. The computer reproduction of chromatograms manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.8.4 If peak detection is prevented by the presence of interferences, further clean-up may be required.

7.8.5 Standards must be injected at the intervals no more than every 20 samples; however, it is recommended that no more than 10 samples be analyzed between CCVs. The calibration factor for each CCV must not exceed the default criteria of ± 15% difference (8082) when compared to the initial calibration and criteria of ± 20 % difference for 8082A. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary. The instrument must be in-control before proceeding with sample analysis. All samples that were injected after the last in-control CCV must be re-injected. If routine maintenance does not return the instrument back into control based on the last initial calibration, verify that proper maintenance has been performed and if still out-of-control start a new initial calibration.

8.0 QUALITY CONTROL

8.1 QC Summary

For each analytical batch (20 samples), a MB, LCS, MS and MSD must be analyzed. If a MS/MSD is not performed, either because of insufficient sample volume, or the client specifically requests that a MS and MSD not be performed, then a LCS duplicate (LCD) is extracted. The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps. Also, every sample must be spiked with the surrogates TCX and DCB. These results are tabulated by the QA/QC to generate in-house control limits.

8.1.1 The MB will be examined to determine if contamination is being introduced in the laboratory. The MB, LCS and MS/MSD must be carried through all stages of the sample preparation, cleanup procedures and measurement steps. The batch QC that has gone through the cleanup procedure will be analyzed with the samples and uploaded in to TALs. Note: If only a portion of the samples in the batch required the cleanup procedure, the batch QC will be set to a status of "Acceptable". This data will be available to the client upon request.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Precision will be measured by the reproducibility of the MSs and will be calculated as Relative Percent Difference (RPD). If MSs were not analyzed, reproducibility will be measured using the LCS/LCD. Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 Surrogate compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable. However, if DCB recovery is low or compounds interfere with DCB, then the TCX should be evaluated for acceptance. Proceed with corrective action when both surrogates are outside of control limits.

8.1.5 Each time an analytical sequence is started, the first Aroclor 1660 must be evaluated to determine if the chromatographic system is operating properly. The analyst should consider:

- Do the peaks look normal?
- Is the response obtained comparable to the response from previous calibrations?

Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc.

8.1.6 Required Instrument QC

8.1.6.1 Section 6.0 requires that the %RSD $\leq$ 20% when comparing calibration factors to determine if the initial calibration curve is linear.

8.1.6.2 Section 6.0 sets a limit of $\pm$15% difference (8082) and $\pm$ 20% difference (8082A) when comparing the continuing response of AR1660 versus the initial response. If the limit is exceeded, corrective action must be taken to correct the problem or the sequence must be started over. All samples following the last standard that was in-control must be reanalyzed.

8.1.6.3 Section 6.2 requires the establishment of retention time windows.

8.1.6.4 Section 7.4.2 sets a limit of $\pm$15% difference (8082) and $\pm$ 20% difference (8082A/DoD QSM) when comparing the initial response of the initial linearity to any succeeding standards analyzed during an analytical sequence.

8.1.6.5 Section 7.5.5 requires that all succeeding standards in an analytical sequence must fall within the daily retention time window established by the first standard of the sequence.

8.1.7 For every batch of samples (20 samples = a batch) the analyst must perform a MS, MSD, MB, and LCS. Also, every sample, and QC sample must be spiked with the surrogates.

8.1.7.1 The desired concentrations of the spikes used are as follows:

Compound	Water (ug/mL)	Soil/Sediment (ug/mL)	Waste/Oil (ug/mL)
AR 1660	5.0	5.0	50.0
TCX	0.40	0.40	4.0
DCB	0.40	0.40	4.0

*Surrogates are prepared in separate solutions from the MS compounds. Refer to the SOP for the preparation of standards and QC solutions.

8.1.8 Limits used for spike recoveries are in-house statistically generated control limits, or limits which have been specifically assigned by the client. If statistical data is not available due to lack of data points, default limits of 70-130% are used until enough data points are available. Refer to the backlog for the requested control limits (see also the QAP, when appropriate).

8.1.9 If a recovery is outside of its control limits, the result should first be examined for any possible calculation errors. If that isn't the problem, then the sample should be re-injected. If after re-injecting the sample it is still out-of-control, and the LCS is in-control, the out-of-control recovery may be attributed to the matrix.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources when determining the action to be taken. The out-of-control situation may or may not be caused by more than one problem. The analyst should seek the help of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation and the required corrective action. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, QA personnel or the lab manager's approval.

Listed below are steps to be taken by the analyst when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed;
- document the problem and the action which was taken to correct the problem on a Non-Conformance Memo (NCM);
- document in the NCM that an in-control situation has been achieved;
- receive approval of the section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

Whenever a problem exists, such as insufficient sample to run MSs and MSDs, a NCM is written. It is filed with the report discussing the actions taken to correct and document the problem. The analyst and their section manager decide what to do with this problem, whether it is analytical, sampling, or matrix interference.
Listed below are some suggested courses of action that may be taken to correct out-of-control situations that may occur with the procedure.

8.2.1 Initial Calibration Curve

For external standard calibration, it is required that a minimum 5-point linearity be run initially (Section 6.1). If the percent relative standard deviation (%RSD) of the calibration factor is $\leq$ 20% for the linearity over the working range, linearity through the origin can be assumed, and the average calibration factor (CF) may be used for calculations. Alternatively, if the correlation coefficient is >0.99, linear regression may be used.

Prior to running a new calibration, or preparing new standards, ensure proper maintenance has been performed. Correct the problem, then re-run the calibration curve and determine new response factor.

8.2.2 Initial Calibration Verification (SSV)

The CF of the initial calibration check standard must not vary from the Average CF of the calibration curve by more than $\pm$ 15% (8082) / $\pm$ 20% (8082A) on both columns. Repeat the standard to verify proper preparation. If still non-compliant, prepare a new standard from the original stock, recalibrate with a new standard curve, or prepare new stock and/or working standards.

8.2.3 Continuing Calibration Verification (CCV)

The response factors of the initial calibration check standard must not vary from the Average CF of the calibration curve by more than ± 15% (8082) and ± 20% (8082A) on both columns. If the average % difference criteria is not met, corrective actions must be taken and documented in order to bring the instrument back into control, or a new calibration curve must be analyzed. If any of the target compounds fail the criteria, then the standard is considered to be non-compliant. If the standard is non-compliant, then reanalyze the standard. If the standard is still non-compliant, remove 3-6 inches from the guard column and condition the column for 1-2 hours. If the standard is still non-compliant, then prepare a new standard. If the new standard is non-compliant, then recalibrate the instrument with a new curve. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard than last met the QC criteria must be re-injected. If a CCV is biased high and all associated samples are clean then this failed criteria should be narrated as showing increased sensitivity, and how this might bias data.

8.2.4 Method Blank (MB)

The MB should not contain any target compounds at or above the reporting limit or ≤ 1/2 RL for special project QAPP compliance. If non-compliant, then the MB should be reanalyzed to determine if instrument contamination was the cause. If the MB is still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCM and reanalyze all associated samples containing target compounds at <10x amount found in the MB. If a sample contains target compounds at ≥ 10x amount found in the MB, or if target compounds are not detected in the sample, then that sample does not require re-extraction and the results may be reported without qualification. If the samples are outside the extraction holding time, then contact the project manager using an NCM. If samples are re-extracted beyond the required holding time, results for both analyses will be reported.

8.2.5 Laboratory Control Sample (LCS)

The recoveries for the spiked compounds must be within statistical limits, or otherwise specified in project or program limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze the LCS to determine if instrumental conditions or analytical preparation was the cause. If the LCS is still non-compliant notify the Project Manager using an NCM, to determine if re-extraction is required of all associated samples. If samples are re-extracted beyond the required holding time, results for both analyses will be reported. Note: State specific or program specific acceptance criteria may be required, check the Project/Job notes in LIMs to determine if any custom limits are applicable.

8.2.6 Laboratory Control Sample Duplicate (LCD)

The LCD must meet all control limits as LCS in addition to limits set for precision (same corrective action as the LCS).

8.2.7 Matrix Spike (MS)

The recoveries for the spiked compounds should be within statistical control limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, and the associated LCSs are within advisory limits, then sample matrix effects are the most likely cause.

8.2.8 Matrix Spike Duplicate (MSD)

See MS for recoveries. RPDs should be within statistical limits. If non-compliant, check calculations for documentable errors. Check unspiked sample results and surrogate recoveries for indications of matrix effects. If significant differences exist between the MS and MSD, reanalysis of the sample spikes may be necessary.

8.2.9 Surrogate Spike

Both recoveries for the surrogate(s) should be within control limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, and the surrogate recoveries in the MB and LCSs are within statistical limits, then sample matrix effects are the most likely cause. Any samples with both surrogate recoveries outside statistical limits with no visible chromatographic cause should be reanalyzed to determine if an injection error was the cause for the out-of-control recovery. If the surrogate recoveries in the associated MB and LCS are not within advisory limits, and the samples are within the extraction holding time, then re-extract and reanalyze all associated samples. If the samples are outside the extraction holding time, then contact the project manager using an NCM. If samples are re-extracted beyond the required holding time, results for both analyses will be reported.

8.2.10 Retention Time Windows

Evaluate the data for usability based on a comparison with the standards run during the analytical sequence. Also, consider the retention times for the surrogates and spiked compounds analyzed before and after the sample in question.

Initial retention time windows must be established following the procedure described in Section 6.2. Daily windows are then established as in Section 6.2.5 at the beginning of each analytical sequence. If a continuing standard is outside of their daily windows, corrective actions must be taken before continuing. Check the septa first and make sure it is tight before any other possibilities are investigated. Check the press connectors, discoloration of the guard column or check the carrier gas and its connection to the instrument for possible leaks.

Perhaps baking the detector at 350°C for about an hour or two will help.

IF AT ANY TIME THE ANALYST IS UNCERTAIN AS TO WHAT ACTIONS TO TAKE OR HOW TO PERFORM SUGGESTED MAINTENANCE, PLEASE SEE YOUR SECTION MANAGER. IF SOMETHING UNUSUAL IS HAPPENING THAT IS NOT DESCRIBED IN THIS SECTION, SEE YOUR SECTION MANAGER. COSTLY (AS IN MONEY AND TIME) DAMAGE CAN RESULT TO THE INSTRUMENT, DETECTOR, OR COLUMN IF MAINTENANCE IS NOT PERFORMED CORRECTLY.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Concentration (ppb) $= \frac{(A_1)(V_t)(Df)}{[(V_i)(V_s)(D)][CF]}$

Where:
A_1 = response for the analyte in the sample (area or height)
V_i = volume of extract injected (mL).
Df = dilution factor (if applicable)
V_t = volume of total extract (mL).
V_s = volume/weight of sample extracted
D = (100 - % Moisture) / 100
CF = Average calibration factor from the initial calibration curve

9.2 %RSD $= \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$

Where,
$$\text{Standard Deviation} = \left| \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$$

Where,
x_1 = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

9.3 % Difference $= \frac{\text{Avg. CF} - \text{CF}}{\text{Avg. CF}} \times 100$

Where:
Avg CF = Average CF from initial linearity
CF = CF from the analysis of the verification standard

9.4 Surrogate Percent Recovery $= \frac{Q_d}{Q_a} \times 100$

Where:
Q_d = Quantity determined by analysis
Q_a = Quantity added to sample/blank

9.5 Spike Recovery $= \frac{SR}{SA} \times 100$

Where:
SR = Spike result
SA = Spike added

9.6 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.7 Precision: $\%D = \frac{|B_1 - B_2|}{\text{Mean of } B_1 \,\&\, B_2} \times 100$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCD)

9.8 Instrument Run-Logs

The analysis of samples and standards is documented electronically within each instrument-specific run log in Chrom, and must be printed for each day's analysis (Attachment 3). All sample information summary sheets must be bound in chronological order (real-time) in the instrument's designated logbook.

9.9 Traceability of Standards

Upon receipt or preparation, each standard is entered into LIMS and is issued a unique ID# based upon the type and sequential order in which the item was received. Further information includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., **XXXX** Stock Solution – LCS/MS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

9.10 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies and in accordance with the TestAmerica corporate procedure CA-T-P-003, *Reporting Results for Methods that Require Second-Column Confirmation* (Attachment 7). The analysts transfer the data into the TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Sample vials are placed in the drum labeled "Flammable vials" for disposal, unless they have a high known or historical PCB concentration.
- Expired primary and working PCB standards and sample extracts that have a known or historically high concentration of PCB will be placed in the waste drum labeled "PCB waste".
- Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Working Standard Solutions and Concentrations – ICAL Form 6
Table 2. Example: Analytical Sequence
Attachment 1. Example: Compound List, Reporting Limits and Statistical Control Limits
Attachment 2. TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points'
Attachment 3. Example: Worklist Run Log Report / Maintenance Log
Attachment 4. Example: Data Review Checklist
Attachment 5. TestAmerica Corporate SOP, CA-Q-S-002, Acceptable Manual Integration Practices
Attachment 6: TestAmerica Corporate Procedure, CA-T-P-003, Reporting Results for Methods that Require Second-Column Confirmation.

14.0 REVISION HISTORY

- Revision 16 updated on 09/20/13
- Annual Review
- Remove reference to the DoD QSM criteria
- Section 5.3 was updated to reflect current standard preparation details.
- Section 6.1.3 was updated to include SOP Change Form details.

Table 1.

Working Standard Solutions and Concentrations / ICAL Form 6

Compound	Intermediate Standard Volume (mLs)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Aroclors-1016/1260	0.8	2.0	5.0	10	7.5	10
Surrogates DCB/TCX	0.2	0.4	1.0	2.0	1.5	2.0
Final Volume	100	100	100	100	50	50

Compound	Approximate Concentrations (ng/uL)					
	Level 1	Level 2	Level 3[1]	Level 4[2]	Level 5	Level 6
Aroclor-1016	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1260	0.040	0.100	0.250	0.500	0.750	1.00
Surrogates	0.004	0.008	0.020	0.040	0.060	0.080
Aroclor-1221	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1232	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1242	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1248	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1254	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1262	0.040	0.100	0.250	0.500	0.750	1.00
Aroclor-1268	0.040	0.100	0.250	0.500	0.750	1.00

These AR1016/AR1260 solution has the surrogates DCB and TCX in them. Aroclor 1016 and 1260 are prepared in the same solution and are referred to as AR1660. Unless otherwise specified in the project or job notes, only AR1660 needs to be analyzed at a minimum of 5 levels to establish linearity. A single point standard is required for the other Aroclors, at the mid-level range (0.50 ng/uL).

[1] Concentration of the SSV-3.
[2] Concentration of the CCV-4.

TestAmerica Laboratories
Initial Calibration Report

Method: \\Chi-svr07\ChromData\GC47-48\20130828-18721.b\8082__47-48.m
Instrument: INST47-48 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: Falcon Last Modified: 30-Aug-2013 09:57:31
No.Compounds:12

Initial Calibration Batches

Ical Batch: \\Chi-svr07\ChromData\GC47-48\20130828-18721.b
Inj Date : 28-Aug-2013 15:11:59, Sublist: chrom-8082__47-48*sub1

Limit Group: GC_PCB_ICAL

Detector 1: Ch-A

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
$ 12 Tetrachloro-m-xylene	6697147	6108200	5698637	5399689	5184417	5054258		5690391		Avg 11
3 PCB-1221-1				62970				62970		Avg 0
3 PCB-1221-2				45636				45636		Avg 0
3 PCB-1221-3				147516				147516		Avg 0
4 PCB-1232-1				122548				122548		Avg 0
4 PCB-1232-2				92180				92180		Avg 0
4 PCB-1232-3				197262				197262		Avg 0
4 PCB-1232-4				92260				92260		Avg 0
4 PCB-1232-5				67580				67580		Avg 0
2 PCB-1016-1	252839	235956	208657	196175	179351	171513		207415		Avg 15
2 PCB-1016-2	202689	192171	171554	163022	149074	143274		170297		Avg 14
2 PCB-1016-3	540438	524024	473446	459143	425997	416511		473260		Avg 11
2 PCB-1016-4	174253	187341	167542	160793	148165	145110		163867		Avg 9.8
2 PCB-1016-5	251992	236723	211884	201327	186587	179867		211397		Avg 13
5 PCB-1242-1				128612				128612		Avg 0
5 PCB-1242-2				361440				361440		Avg 0
5 PCB-1242-3				126790				126790		Avg 0
5 PCB-1242-4				167922				167922		Avg 0
5 PCB-1242-5				220078				220078		Avg 0
6 PCB-1248-1				239860				239860		Avg 0
6 PCB-1248-2				240134				240134		Avg 0
6 PCB-1248-3				362186				362186		Avg 0
6 PCB-1248-4				256040				256040		Avg 0
6 PCB-1248-5				124458				124458		Avg 0
7 PCB-1254-1				284036				284036		Avg 0
7 PCB-1254-2				391749				391749		Avg 0
7 PCB-1254-3				501198				501198		Avg 0
7 PCB-1254-4				464863				464863		Avg 0
7 PCB-1254-5				473803				473803		Avg 0
8 PCB-1260-1	501394	446534	392869	374070	347523	339459		400308		Avg 16
8 PCB-1260-2	574029	520876	467287	449072	416869	407897		472672		Avg 14
8 PCB-1260-3	657346	597341	540104	524625	489525	482033		548495		Avg 12
8 PCB-1260-4	554283	504233	453713	438765	408300	404156		460575		Avg 13
8 PCB-1260-5	326718	293865	262155	246070	228887	223122		263469		Avg 15
9 PCB-1262-1				344580				344580		Avg 0
9 PCB-1262-2				496252				496252		Avg 0
9 PCB-1262-3				435574				435574		Avg 0
9 PCB-1262-4				918552				918552		Avg 0
10 PCB-1268-1				190628				190628		Avg 0
10 PCB-1268-2				237236				237236		Avg 0
10 PCB-1268-3				1202846				1202846		Avg 0
10 PCB-1268-4				865142				865142		Avg 0
10 PCB-1268-5				2392390				2392390		Avg 0
$ 1 DCB Decachlorobiphenyl	10190750	8980750	8075350	7463875	7037717	6967088		8119255		Avg 16

Method: \\Chi-svr07\ChromData\GC47-48\20130828-18721.b\8082__47-48.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2

Detector 2: Ch=B

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
$ 12 Tetrachloro-m-xylene	4090132	3680294	3547931	3401469	3359314	3372459		3575266		Avg 7.9
3 PCB-1221-1				43756				43756		Avg 0
3 PCB-1221-2				31688				31688		Avg 0
3 PCB-1221-3				98720				98720		Avg 0
4 PCB-1232-1				85328				85328		Avg 0
4 PCB-1232-2				66324				66324		Avg 0
4 PCB-1232-3				135088				135088		Avg 0
4 PCB-1232-4				69652				69652		Avg 0
4 PCB-1232-5				58602				58602		Avg 0
2 PCB-1016-1	101942	94681	86267	79287	73559	70943		84447		Avg 14
2 PCB-1016-2	381399	341902	317159	313004	302544	299749		325960		Avg 9.5
2 PCB-1016-3	195070	177988	159833	154398	145384	141402		162346		Avg 13
2 PCB-1016-4	137550	125827	118092	114267	107283	106054		118179		Avg 10
2 PCB-1016-5	173406	158546	144020	137452	130352	125727		144917		Avg 12
5 PCB-1242-1				108778				108778		Avg 0
5 PCB-1242-2				237474				237474		Avg 0
5 PCB-1242-3				118614				118614		Avg 0
5 PCB-1242-4				110366				110366		Avg 0
5 PCB-1242-5				127772				127772		Avg 0
6 PCB-1248-1				126792				126792		Avg 0
6 PCB-1248-2				178202				178202		Avg 0
6 PCB-1248-3				171492				171492		Avg 0
6 PCB-1248-4				226798				226798		Avg 0
6 PCB-1248-5				199844				199844		Avg 0
7 PCB-1254-1				206289				206289		Avg 0
7 PCB-1254-2				244419				244419		Avg 0
7 PCB-1254-3				386692				386692		Avg 0
7 PCB-1254-4				171821				171821		Avg 0
7 PCB-1254-5				239597				239597		Avg 0
8 PCB-1260-1	380254	337301	300203	291789	279445	274298		310548		Avg 13
8 PCB-1260-2	452291	407062	367359	363926	345580	340176		379399		Avg 11
8 PCB-1260-3	299153	265129	242179	235207	221855	219257		247130		Avg 12
8 PCB-1260-4	447983	402271	369550	366347	352924	354680		382292		Avg 9.6
8 PCB-1260-5	219970	197649	176036	167169	157203	153470		178583		Avg 14
9 PCB-1262-1				226146				226146		Avg 0
9 PCB-1262-2				400776				400776		Avg 0
9 PCB-1262-3				376726				376726		Avg 0
9 PCB-1262-4				772826				772826		Avg 0
10 PCB-1268-1				202504				202504		Avg 0
10 PCB-1268-2				870988				870988		Avg 0
10 PCB-1268-3				808722				808722		Avg 0
10 PCB-1268-4				2653580				2653580		Avg 0
10 PCB-1268-5				488574				488574		Avg 0
$ 1 DCB Decachlorobiphenyl	8514250	7624250	6907000	6496125	6378217	6311725		7038594		Avg 12

Table 2.

Example: Analytical Sequence

Quantitation & Confirmation of Aroclors			
1.	AR 1660-6	1.	AR 1660-6
2.	AR 1660-5	2.	AR 1660-5
3.	AR 1660-4	3.	AR 1660-4
4.	AR 1660-3	4.	AR 1660-3
5.	AR 1660-2	5.	AR 1660-2
6.	AR 1660-1	6.	AR 1660-1
7.	AR 1254-4	7.	AR 1254-4
8.	AR 1248-4	8.	AR 1248-4
9.	AR 1242-4	9.	AR 1242-4
10.	AR 1232-4	10.	AR 1232-4
11.	AR 1221-4	11.	AR 1221-4
12.	AR 1268-4	12.	AR 1268-4
13.	AR 1262-4	13.	AR 1262-4
14.	PEM Std	14.	PEM Std
15	AR1660 SSV-3	15	AR1660 SSV-3
16	AR 1660-CCV-4	16	AR 1660-CCV-4
17-26.	Samples	17-26.	Samples
27.	AR1660-CCV-4	27.	AR1660-CCV-4
28-37.	Samples	28-37.	Samples
38.	AR1660 CCV-4	38.	AR1660 CCV-4

NOTE: AR1660 linearity is sufficient for initial calibration of all Aroclors since they include all congeners present in the different regulated Aroclors. Special project or program requirements may require additional Aroclor linearities and CCVs performed. The analyst's backlog will alert staff when special project requirements need to be applied to samples.

Attachment 1.

Example: Compound List, Reporting Limits and Statistical Control Limits (024-001 to 024-002)

MDL W_LVI	Polychlorinated Biphenyls (PCBs)	8082A

Analyte Description	CAS Number	RL	LOD	MDL	Units	LCS LCL	LCS UCL	RPD	MS LCL	MS UCL	RPD	Surr LCL	Surr. UCL	Units
PCB-1016	12674-11-2	0.4	0.2	0.067	ug/L	64	110	20	64	110	20			%
PCB-1221	11104-28-2	0.4	0.2	0.2	ug/L									%
PCB-1232	11141-16-5	0.4	0.2	0.2	ug/L									%
PCB-1242	53469-21-9	0.4	0.2	0.2	ug/L	60	120	20	60	120	20			%
PCB-1248	12672-29-6	0.4	0.2	0.2	ug/L	60	120	20	60	120	20			%
PCB-1254	11097-69-1	0.4	0.2	0.2	ug/L	60	120	20	60	120	20			%
PCB-1260	11096-82-5	0.4	0.2	0.07	ug/L	51	110	20	51	110	20			%
PCB-1262	37324-23-5	0.4	0.2	0.2	ug/L									%
PCB-1268	11100-14-4	0.40	0.20	0.20	ug/L									%
Tetrachloro-m-xylene	877-09-8				ug/L							50	120	%
DCB Decachlorobiphenyl	2051-24-3				ug/L							29	126	%

MDL W_LVI	Liquid-Liquid Extraction (Separatory Funnel)	3510C_LVI

(024-001)

MDL S GC Extracatables	Polychlorinated Biphenyls (PCBs)	8082

Analyte Description	CAS Number	RL	LOD	MDL	Units	LCS LCL	LCS UCL	RPD	MS LCL	MS UCL	RPD	Surr LCL	Surr UCL	Units
PCB-1016	12674-11-2	16.7	8.3	5.9	ug/Kg	59	110	30	59	110	30			%
PCB-1221	11104-28-2	16.7	8.3	7.34	ug/Kg									%
PCB-1232	11141-16-5	16.7	8.3	7.27	ug/Kg									%
PCB-1242	53469-21-9	16.7	8.3	5.48	ug/Kg	50	120	30	50	120	30			%
PCB-1248	12672-29-6	16.7	8.3	6.57	ug/Kg	50	120	30	50	120	30			%
PCB-1254	11097-69-1	16.7	8.3	3.6	ug/Kg	50	120	30	50	120	30			%
PCB-1260	11096-82-5	16.7	8.3	8.19	ug/Kg	69	120	30	69	120	30			%
Tetrachloro-m-xylene	877-09-8				ug/Kg							50	116	%
DCB Decachlorobiphenyl	2051-24-3				ug/Kg							48	142	%

MDL S GC Extracatables	Automated Soxhlet Extraction	3541

(024-002)

Attachment 2.

TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (025-001 to 025-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

06/18/2012		12/21/2012	
Technical Services Director	Date	Quality and EH&S Director	Date

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(625-001)

1.0 PURPOSE

This policy describes TestAmerica Laboratories, Inc. requirements for the determination of the number of points and removal of points from calibration curves.

2.0 SCOPE

This policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP), then that requirement will be used for the defined procedure.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the DQOs for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a single point at the reporting level.

Examples:

A. Required to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by HPLC-UV at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

   The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the DQOs will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in the laboratory for meeting a regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A 5-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where the technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS's used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the QA Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of points from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24-hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internal standards show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (All analytes in spike mix must show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection.)

(025-003)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers), the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.
- Revision 2.1, dated 11 June 2012.
  - General clarifications – no procedural changes.

Attachment 3.

Example: Worklist Run Log Report / Maintenance Log (026-001 to 026-002)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 091913 Worklist Num: 19076
Instrument: INST23-24 Method: 8082_23-24
Batch Directory: \\Chi-svr07\ChromData\GC23-24\20130919-19076.b
Anaylysis Type: SemiVOA Creator: Orf, Gene M
Inj Volume: 1.00 Inj Vol Units: ul

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
CCV AR1660	500-0019076-001	CCV		19-Sep-2013 08:49:29	090613_172.D		1.0		sv
MB 500-202712/1-A	500-0019076-002	MB		19-Sep-2013 09:03:25	090613_173.D		1.0		sv
LCS 500-202712/3-A	500-0019076-003	LCS		19-Sep-2013 09:17:25	090613_174.D		1.0		sv
500-62486-E-1-F	500-0019076-004	Client		19-Sep-2013 09:31:25	090613_175.D		1.0	SSI2013-SB-479-0	sv
500-62486-E-4-D	500-0019076-005	Client		19-Sep-2013 09:45:38	090613_176.D		1.0	SSI2013-SB-469-0	sv
500-62486-E-5-G	500-0019076-006	Client		19-Sep-2013 09:59:38	090613_177.D		1.0	SSI2013-SB-469-0-D	sv
500-62563-F-1-B	500-0019076-007	Client		19-Sep-2013 10:13:37	090613_178.D		1.0	SSI2013-SB-489-0	sv
500-62642-E-2-C	500-0019076-008	Client		19-Sep-2013 10:27:36	090613_179.D		1.0	SSI2013-SB-499-4	sv
500-62642-E-5-C	500-0019076-009	Client		19-Sep-2013 10:41:36	090613_180.D		1.0	SSI2013-SB-503-4	sv
500-62642-F-6-C	500-0019076-010	Client		19-Sep-2013 10:55:48	090613_181.D		1.0	SSI2013-SB-500-4	sv
500-62642-F-7-B	500-0019076-011	Client		19-Sep-2013 11:09:48	090613_182.D		1.0	SSI2013-SB-500-10	sv
CCV AR1660	500-0019076-012	CCV		19-Sep-2013 11:23:47	090613_183.D		1.0		sv
500-62723-F-1-C	500-0019076-013	Client		19-Sep-2013 11:37:46	090613_184.D		1.0	SSI2013-SB-501-0	sv
500-62723-F-3-I	500-0019076-014	Client		19-Sep-2013 11:51:44	090613_185.D		1.0	SSI2013-SB-501-4D	sv
500-62723-F-3-L MS	500-0019076-015	MS		19-Sep-2013 12:05:52	090613_186.D		1.0	SSI2013-SB-501-4D	sv
500-62723-F-3-M MSD	500-0019076-016	MSD		19-Sep-2013 12:19:48	090613_187.D		1.0	SSI2013-SB-501-4D	sv
500-62723-F-4-C	500-0019076-017	Client		19-Sep-2013 12:33:43	090613_188.D		1.0	SSI2013-SB-501-4	sv
500-62816-F-1-B	500-0019076-018	Client		19-Sep-2013 12:47:42	090613_189.D		1.0	SSI2013-SB-510-0	sv
500-62816-F-2-B	500-0019076-019	Client		19-Sep-2013 13:01:40	090613_190.D		1.0	SSI2013-SB-510A-0	sv
CCV AR1660	500-0019076-020	CCV		19-Sep-2013 13:15:52	090613_191.D		1.0		sv
500-62723-F-1-C	500-0019076-021	Client		19-Sep-2013 13:43:52	090613_193.D		20.0	SSI2013-SB-501-0	sv
CCV AR1660	500-0019076-022	CCV		19-Sep-2013 13:57:52	090613_194.D		1.0		sv

Hexane: Lot #41837

(026-001)

TestAmerica Chicago
GC Extractables Instrument Maintenance Log

Page No.:_____
Instrument No.: 41 / 42

Routine/Informal Maintenance includes: Glass Y-connectors checked for leaks; periodic system check for leaks, frayed wires and a general overview; flows checked

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Reviewer Signature:_____ Date:_____

Attachment 4.

Example: Data Review Checklist
(027-001 to 027-003)

GC/HPLC Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
REPORTS:		
p-Flag report reviewed and accepted?		
Retention Time Study reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: <20% RSD; for 608 and 610 < 10% RSD		
Linear or Quadratic Regression curve fit for all >20% RSD R^2 >0.990		
Plots for all Linear Regressions reviewed?		
conc-intercept < RL; (<1/2 RL QAPP specific); <30% Error (Chrom: Details of Calibration per Analyte)		
Co-eluting peaks reviewed and accepted?		
All manual integrations reviewed found to be necessary, performed correctly and have documented reasons?		
Method:		
Method locked in Chrom?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct and locked in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 85-115% (15%D) criteria? 8015C; 8082A; 8081B (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date_____

Secondary Reviewer: _____ Date_____

(027-001)

TestAmerica Chicago

GC EXTRACTABLES / HPLC DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Analytical Method: a) GC _____ b) HPLC _____ Instrument (Primary / Confirmation):_____

Extraction Method: a) SW 3510 b) SW 3541 c) SW 3550 d) SW 3580 e) Other (_____)

Matrix: WATER / SOIL / SPLP-TCLP / Waste / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: _____ _____ _____ _____ _____
Import Batch Review: Is the Calibration ID Correct? Y N Report Status of Primary / Secondary correct? Y N CCV's are correct and properly linked to samples? Y N Calculation of all QC complete? Y N			
View All Data Review: *Samples:* Results reported for all samples? Y N Dilutions / Re-analyses are correctly represented? Y N NA Are flags correctly assigned? Y N *QA Summary: QC Association Summary* All required dilutions/re-analyses present? Y N All Samples linked to prep batch? Y N All Samples linked to batch QC? Y N *Lab QC:* All QC present and correctly represented? Y N *Surrogate*: All samples/QC present and correct? Y N			
Form 1 / View All Data: IF **original** and **re-analyses** are to be reported in LIMS: Suffix Used: Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Sample # Original Dilution Comments
FORM 2 / View All Data: Surrogate Recoveries within Limits? Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3 / View All Data: MS/MSD Recoveries Acceptable Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3 / View All Data: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

(027-002)

TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Criteria Met QAPP specific MB < ½ RL _____ NCM Ref Number: _____			
FORM 4: 1) Primary and Secondary Column info present? Yes No NA 2) All Samples Present Yes No NA 3) Sulfur and GPC clean-ups identified? Yes No NA 4) Header Information Correct? Yes No NA NCM Ref Number: _____			
FORM 8: Retention Time Window Criteria Met? Yes No NCM Ref Number: _____			
Form 10: 1) Retention Time Window Criteria Met? Yes No NA 2) All hits present/Concentrations Correct? Yes No NA 3) Are hits < 40%D between primary & secondary columns? Yes No (NCM Ref No.: _____)			
RAW DATA: 1) Raw Data Verified and Complete? Yes No NA			
2) Raw Data Matches Forms? Yes No NA			
3) Sample info complete-% solids, Smp.Wt, F.V.?			
4) Prep/Extraction Data info verified in Raw Data?			
Form 6: Initial Calibration Criteria met and Complete? Samples linked to ICAL? _____			
Form 7: CCV criteria met method criteria Samples linked to CCV? _____ NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Blank / LCS / LCSD Yes No 2) MS/MSD Yes No NA 3) MRL Check Required: Yes No Limits Applied: _____			
Manual Integration reports (before and afters) present and reasons correctly documented and approved?			
Prep / Sample Summary pages Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required / Properly Documented? Yes No			
Manual Calculation of On Column result: Peak Height or Area of Analyte Average Peak Height or Area from ICAL			Sample : _____ Compound: _____
JOB STATUS: 1) Primary Reviewer/Analyst: 1st / 2nd Level			
2) Secondary Reviewer/Supervisor: Lab Complete			
Additional Comments:			

(027-003)

Attachment 5.

TestAmerica Corporate SOP, CA-Q-S-002,
'Acceptable Manual Integration Practices'
(028-001 to 028-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

08/01/2012	08/01/2012
Raymond J. Frederici Quality and EH&S Director	Date	Larry Penfold Quality Compliance Director	Date
08/01/2012	
Verl D. Preston Quality Assessment Director	Date		

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1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

4.5 **Baseline:** The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 **Peak:** An increase in signal from baseline to a maximum and then back to baseline.

4.7 **Coelution:** The concurrent elution of two or more compounds to the detector at the same time.

4.8 **Elution:** The process of movement of compounds from the chromatographic system.

4.9 **Carry-over:** Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 **Peak Tailing:** Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 **Interference Peaks or Fused Peaks:** Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important considerations in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters is necessary to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibrations standards, QC standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. This

process for before & after chromatograms is also required for OVAP-approved laboratories, unless instructed otherwise.

5.4.7.1 Re-integration technique marked, if available, on the data system.

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.

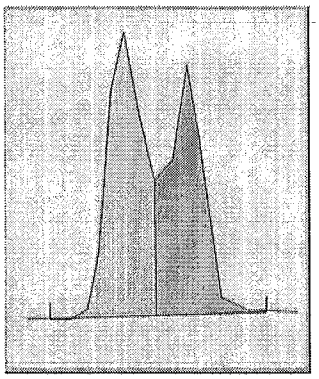
- Expanded section on data miner software to include Chrom Audit Miner
- Included reference to data miner training presentation.

- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples

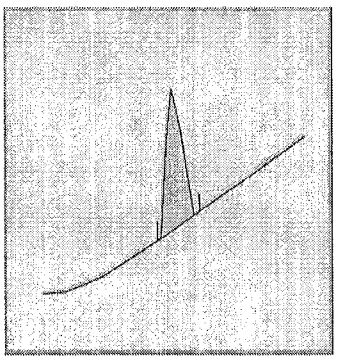
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

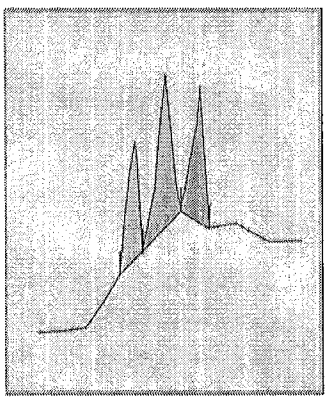
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.

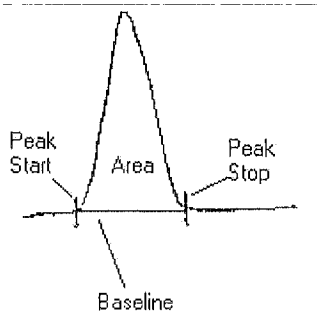
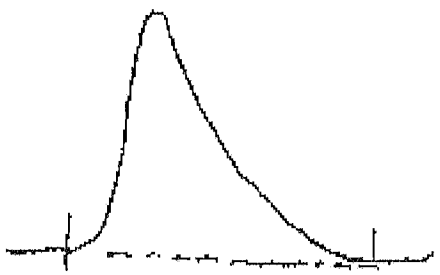


Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.



Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.



Ideal peak (Gaussian Shape) The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

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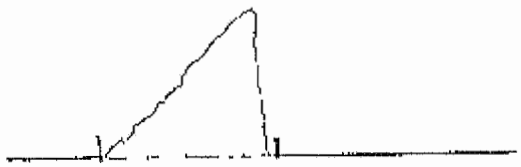
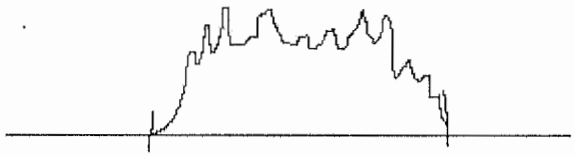
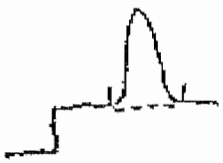
Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	 Negative Dip  Suden Rise

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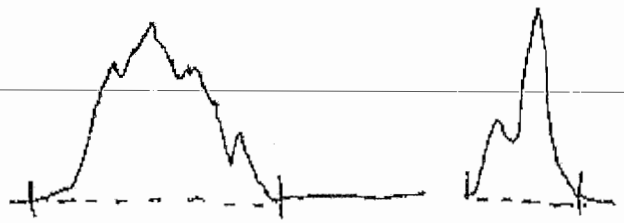
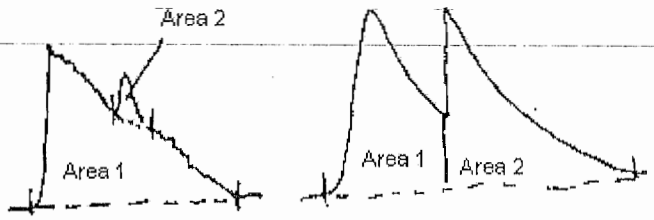
Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

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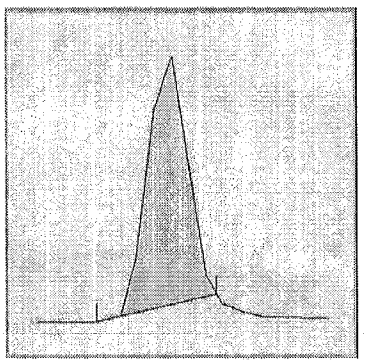
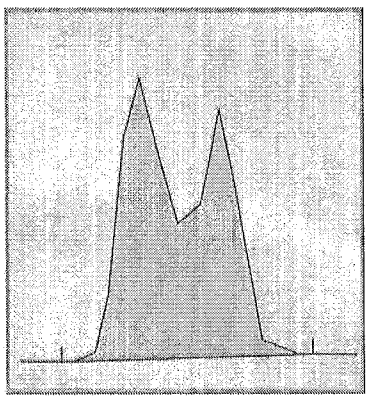
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

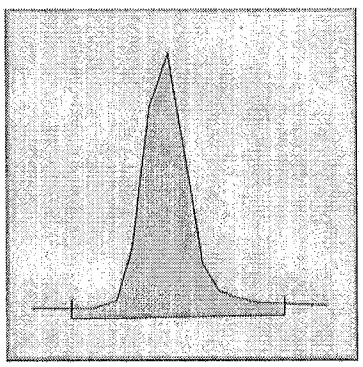
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.



Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



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Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.

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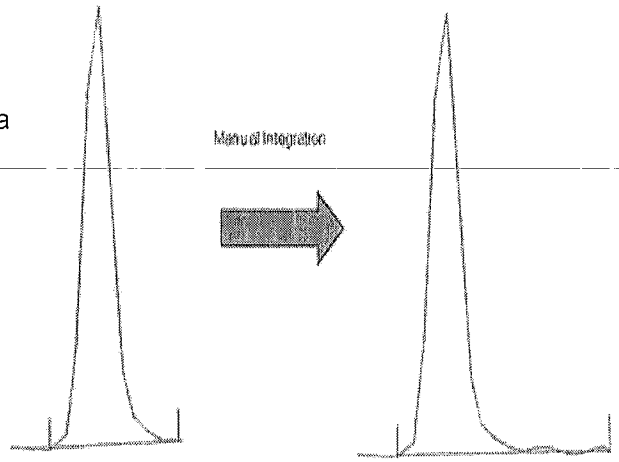
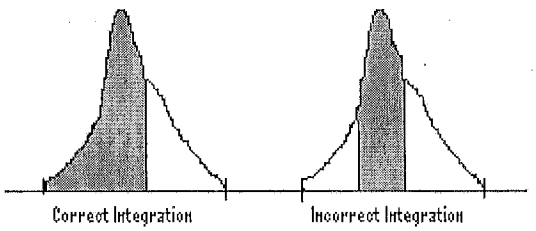


An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 6.

TestAmerica Corporate Procedure, CA-T-P-003,
Reporting Results for Methods that Require Second-Column Confirmation
(029-001 to 029-003)

Reporting Results for Methods that Require Second-Column Confirmation

Approvals (Signature/Date):

Technical Services Director	06/18/2012 Date	Quality and EH&S Director	12/21/2012 Date

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(029-001)

1.0 PURPOSE

This policy describes a standardized approach to reporting results for methods that require second-column confirmation, such as EPA SW-846 8000 series GC methods. This policy is designed to be in compliance with EPA methods 8000B and 8000C as well as EPA 600 series methods and is intended to be followed in the absence of QAPP or regulatory guidance to the contrary (e.g., DOD, DOE, and CLP).

2.0 SCOPE

This policy applies to all methods that require second-column (qualitative and quantitative) confirmations. This procedure is not applicable to qualitative only confirmations, such as Methods 8330 or 8330A, which report only from the primary column.

3.0 SAFETY

Refer to Laboratory-specific method SOPs.

4.0 DEFINITIONS

None.

5.0 PROCEDURE

5.1 The laboratory may designate a primary column for each analyte (in which case the second column is the confirmation column for each analyte), or may report the lower result from the two columns. The same column does not have to be primary for every analyte. Both columns are intended to be calibrated and meet calibration verification requirements.

5.2 Whichever approach is used, QC must be assessed and reported in the same way as the samples (i.e., report from the primary column or report the lower number).

5.3 40% RPD Criterion

5.3.1 When using the primary column option, if the RPD for the dual column results is $\leq$ 40%, then the result from the primary column is normally reported. The exception is situations where a chromatographic interferent is evident in the primary column chromatogram that is not present in the confirmation column chromatogram.

5.3.2 If using the primary column option and the RPD for the dual column results is > 40%, then the lower value is reported. (A RPD of >40% is clear evidence of chromatographic interference on the higher value). A case narrative comment or footnote discussing the RPD failure must be included in the final report.

5.4 Calibration Verification Criterion – Primary Column Option

5.4.1 If the CCV recovery for the primary column is within acceptance limits and the analyte is confirmed on the secondary column, but the secondary column is outside acceptance limits then results from the primary column may be reported with a case narrative comment or footnote.

5.4.2 If the CCV recovery for the confirmation column is within acceptance limits and the recovery for the primary column is in the range of +15% to +40%, then results may be reported from the confirmation column with a case narrative comment or footnote.

5.4.3 If the CCV recovery is below acceptance limits for the primary column, or the secondary column is below acceptance limits and the analyte is not confirmed, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.4.4 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.5 Calibration Verification Criterion – Lower Result Option

5.5.1 If the CCV recovery for the column with the lower result is within acceptance limits and the analyte is confirmed on the secondary column but the secondary column is outside acceptance limits, then results from the lower column may be reported with a case narrative or footnote.

5.5.2 If the CCV recovery for the higher result is within acceptance limits but the lower result column fails acceptance limits and the RPD is < 15%, then the higher result may be reported with a case narrative or footnote.

5.5.3 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving second-column confirmation are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 6 November 2007
  - Initial Release.
- Revision 1, dated 23 June 2009
  - No Changes
- Revision 2, dated 18 June 2012
  - Added quantitative column confirmation
  - Added limits for CCV failures on confirmation columns; and clarified language.

TITLE: GAS CHROMATOGRAPHY - SEMI-VOLATILES Herbicides by SW-846 Method 8151A

Approvals (Signature/Date):

Sharon Werner 7/31/13 Sharon Werner — Date GC Chemist	
Gary L. Rynkar 7/31/13 Gary L. Rynkar — Date Supervisor GC Extr./HPLC	Mark A Storm 7/31/13 Mark Storm — Date Env. Health & Safety Coor.
Terese A. Preston 7/31/13 Terese A. Preston — Date Quality Assurance Manager	Michael J. Healy 7/31/13 Michael J. Healy — Date Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the analysis of Phenoxy Acid Herbicides in wastewater, soil and sediment extracts. This SOP has been written using SW-846, 3rd Edition, Methods 8000B/C and 8151A as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Refer to Table 1 for the compound listing and their associated reporting limits.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

Chlorinated Phenoxy Acid Herbicides are determined by gas chromatography (GC). Because these compounds may occur in various forms (such as acids, salts & esters), a hydrolysis step is included to permit determination of the active part of the herbicide.

Chlorinated phenoxy acids and their esters are extracted from the acidified sample with ethyl ether. The extracts are hydrolyzed and the extraneous material is removed by a solvent wash. The acids are converted to methyl esters and are determined by a gas chromatograph.

2.0 INTERFERENCES

Organic acids, especially chlorinated acids, cause the most direct interference. Phenols, including chlorophenols, may also interfere. Alkaline hydrolysis and subsequent extractions eliminate many of the predominant chlorinated pesticides. Because the herbicides react readily with alkaline substances, loss may occur if there is alkaline contact at any time except in the controlled alkaline hydrolysis step. The use of acid rinsed glassware and acidified sodium sulfate (Na_2SO_4) will avoid this problem.

2.1 Glassware

All glassware/equipment used in the preparation of standards (volumetric flasks, pipettes, beakers, eppendorf tips) are rinsed well with the appropriate solvent and allowed to dry before use. After completion of standard preparation, unused amounts of standards are properly disposed of, and all glassware/equipment rinsed well with the appropriate solvent. Rinse/excess solvent is properly collected and disposed of. These procedures should eliminate positive interferences from these sources.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- ECDs contain a radioactive source. Special regulations apply.
- Interior parts of GC's can be very hot. Care should be taken if adjusting instrument.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of with six months.**
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Supplies

- Autosampler vials (2 mL) with Teflon-lined caps
- Pipets - Class A volumetric
- Transfer pipets
- Volumetric flasks - Class A
- Gases: Hydrogen (carrier) and Nitrogen (make-up)

4.2 Gas Chromatographic Systems

- HP 6890 GC equipped with dual electron capture detectors (ECD - Ni63) and two autosamplers.
- Each GC uses Total Chrom for data acquisition and Chrom for processing data.

4.3 Analytical Columns

- Column 1/signal 0 - Restek-Rtx-CLP Pestides, 30 meter, 0.53 mm ID, 0.5-micron film thickness or equivalent.
- Column 2/signal 1 - Restek-Rtx-CLP Pestides2, 30 meter, 0.53 mm ID, 0.42-micron film thickness or equivalent.
- Restek-Rtx-35, 30 meter, 0.53 mm ID, 0.5-micron film thickness or equivalent.

Signal 0 is designated as the primary column in TALs and signal 1 is designated as the secondary column in TALs. Additional guidance can be found in Attachment 6, the corporate SOP CA-T-P-003, *Reporting Results for Methods That Require Second-Column Confirmation.* Similar columns can be used if all QC criteria can be met.

5.0 REAGENTS AND STANDARDS

The preparation of all standards and QC solutions must be properly documented. A unique reagent code is created in the TALs reagent module. The information includes the name of the reagent, the concentration of the reagent, the date prepared, expiration date and the analyst who prepared the reagent. The reagent label used to label each reagent bottle contains the unique TALs code for that reagent bottle, its description, expiration date, prepped/received date, solvent use, prepared by and date created. The label also contains a bar code for scanning into the Chrom worklists, which then transfer to the TALs AD batches upon upload.

Standards must be stored in amber glass containers with Teflon-lined lids at 4 $\pm$ 2°C, protected from the light. Stock standards must be replaced after 1 year or sooner if comparison with a check standard indicates a problem.

5.1 Solvents*

Hexane, Ethyl Ether, Methanol, Acetone - *Pesticide grade or better. Silicic acid, Isooctane, (Trimethylsilyl)diazomethane,

5.2 Quality Control (QC) Solution

The herbicide stock ready solutions are purchased from a vendor such as Chem Service (ICV/SSV) or Ultra (ICAL). All standards must be logged into LIMS. Standards are good for 1 year of opened date or until the manufacturer expiration date, whichever is sooner.

5.2.1 Concentrated Stock Solution – ICV/SSV (Chem Service)

Compound	Concentration (ug/mL)
Dalapon	1000
4-Nitrophenol	1000
Dicamba	1000
Dichlorprop	1000
2,4-D	1000
Pentachlorophenol	1000
2,4,5-TP (Silvex)	1000
2,4,5-T	1000
2,4-DB	1000
Dinoseb	1000
Picloram	1000

5.2.1.1 Intermediate Stock Solution – ICV/SSV (Parent) = Soil Spike

Preparation of Primary Herbicide Intermediate Solution:

The concentrated stock solutions are prepared by adding 4 mL of acetone mix standard to 100 mLs of Methanol in a 100 mL volumetric flask.

Compound	Concentration (ug/mL)
Dalapon	40
4-Nitrophenol	40
Dicamba	40
Dichlorprop	40
2,4-D	40
Pentachlorophenol	40
2,4,5-TP (Silvex)	40
2,4,5-T	40
2,4-DB	40
Dinoseb	40
Picloram	40

This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.1.2 Working Solution – ICV/SSV

Preparation of the 0.4 ng/uL Alternate Source Multicomponent Herbicide Mixed Solutions:

Add 1 mL isooctane, 0.5 mL acetone and 1 mL of hexane to a 10 mL volumetric flask. Accurately add 0.1 mL of the ICV/SSV Intermediate Stock Solution (Soil Spike) to the volumetric with a syringe. Add 100 uL (Trimethylsilyl)diazomethane solution and let it react for 1 hour. Make sure the yellow color persists. Quench the reaction with 0.1 – 0.2 g silicic acid. Dilute to the 10 mL mark with Hexane. Transfer the standard to the sealed amber vial, label and refrigerate until needed.

Compound	Concentration (ug/mL)
Dalapon	0.4
4-Nitrophenol	0.4
Dicamba	0.4
Dichlorprop	0.4
2,4-D	0.4
Pentachlorophenol	0.4
2,4,5-TP (Silvex)	0.4
2,4,5-T	0.4
2,4-DB	0.4
Dinoseb	0.4
Picloram	0.4

This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

* 4-Nitrophenol is analyzed for the water matrix only.

5.2.2 Stock Solution – Spike 10x

Standard materials are purchased in acetone solution form. Standards are purchased from vendors such as ChemService.

The concentrated stock solutions are prepared by adding 4 mL of acetone mix standard to 100 mLs of Methanol in a 100 mL volumetric flask.

Compound	Concentration (ug/mL)
Dalapon	40
Dicamba	40
Dichlorprop	40
2,4-D	40
Pentachlorophenol	40
2,4,5-TP (Silvex)	40
2,4,5-T	40
2,4-DB	40
Dinoseb	40
Picloram	40
4-Nitrophenol	40

This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.2.1 Spike Solution 10x (Soil)

1 mL of the 10x Spike Solution is added to all soil laboratory control sample (LCS) and matrix spikes (MS). Soil sample extracts are routinely diluted 1/10 before analysis. This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.2.2 Spike Solution 1x (Water)

The water spike solution is prepared by adding 10 mL of the 10x soil spike to 100 mLs of Methanol in a 100 mL volumetric flask. The resulting concentrations should be:

Compound	Concentration (ug/mL)
Dalapon	4.0
Dicamba	4.0
Dichlorprop	4.0
2,4-D	4.0
Pentachlorophenol	4.0
2,4,5-TP (Silvex)	4.0
2,4,5-T	4.0
2,4-DB	4.0
Dinoseb	4.0
Picloram	4.0
4-Nitrophenol	4.0

1 mL of the 1x spike solution is added to the water and TCLP LCSs and matrix spike/matrix spike duplicates (MS/MSD). Water and TCLP sample extracts are analyzed undiluted. This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.3 Stock Surrogate Solution (5,000 ug/mL)

The stock surrogate solution is purchased at a concentration of 5,000 ug/mL from a vendor (usually Ultra) as a certified material. The surrogate compound used for this analysis is 2,4-Dichlorophenylacetic acid (DCAA). This standard is valid for 1 year, unless otherwise noted by the vendor.

5.2.3.1 10x Surrogate Spike Solution

A 1/100 dilution of the stock solution is made using 1.0 mL and diluted it to 100 mL in methanol, resulting in a concentration of 50.0 ug/mL. 1.0 mL of this solution is added to all soil and TCLP samples, LCSs, MS/MSDs, method blank (MB) and TCLP blanks prior to extraction. This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.2.3.2 1x Surrogate Spike Solution

A 1/10 dilution of the 10x Surrogate Spike Solution is made, resulting in a concentration of 5.00 ug/mL. 1.0 mL of this solution is added to all water samples, LCS(s), MS/MSDs and MBs prior to extraction. This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.3 Standards

5.3.1 Concentrated Stock Solution – ICAL (ULTRA)

Compound	Concentration (ug/mL)
Dalapon	100
Dicamba	100
Dichlorprop	100
2,4-D	100
Pentachlorophenol	100
2,4,5-TP (Silvex)	100
2,4,5-T	100
2,4-DB	100
Dinoseb	100
Picloram	100
4-Nitrophenol	100

5.3.1.1 Parent Standard Solution

Preparation of Primary Herbicide/Surrogate Intermediate Solution:

Add 1.0 mL isooctane, 0.5 mL acetone and 1.0 mL of hexane to a 10 mL volumetric flask. Accurately add 1.0 mL of the certified herbicide mixture (ULTRA) and 0.02 mL of the DCAA solution (ULTRA) to the volumetric with a syringe. Add 100 uL (Trimethylsilyl)diazomethane solution and let it react for 1 hour. Make sure the yellow color persists. Quench the reaction with 0.1 – 0.2 g silicic acid. Dilute to the 10 mL mark with Hexane. Transfer the standard to the sealed amber vial, label and refrigerate until needed. (All components in this resulting solution are present at a concentration of 10 ug/mL.)

Compound	Concentration (ng/uL)
Dalapon	10
Dicamba	10
Dichlorprop	10
2,4-D	10
Pentachlorophenol	10
2,4,5-TP (Silvex)	10
2,4,5-T	10
2,4-DB	10
Dinoseb	10
Picloram	10
4-Nitrophenol	10
DCAA	10

This solution is valid for 6 months or sooner (if comparison with check standards indicates a problem) from the date of preparation.

5.3.1.2 Intermediate Standards

The calibration levels of the standards are made from the Parent Standard in Hexane to provide the following concentrations:

Calibration Level	Concentraton (ng/uL)
6	0.500
5	0.400
4	0.300
3	0.200
2	0.100
1	0.050

This solution is valid for 3 months or sooner due to the degradation of Dinoseb (if comparison with check standards indicates a problem) from the date of preparation.

5.3.4 Working Standards

5.3.4.1 0.050 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 50 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

5.3.4.2 0.10 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 100 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

5.3.4.3 0.20 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 200 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

5.3.4.4 0.30 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 300 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

5.3.4.5 0.40 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 400 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

5.3.4.6 0.50 ng/uL Multicomponent Herbicide Mixed Solution and surrogate. This is prepared by adding 500 uL of the Herbicide/surrogate intermediate solution to a 10 mL volumetric flask and diluting to the mark with hexane.

This solution is valid for 2 months from the date of preparation.

6.0 CALIBRATION

6.1 Retention Time Windows

Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of the standard solution throughout the course of a 72 -hour period. Serial injections over less than a 72-hour period result in retention time windows (RTW) that are too tight, the minimum RT window criteria is ± 0.03 min.

Calculate the standard deviation of the three absolute retention times for each single component standard. ± 3X standard deviation of the absolute retention times for each standard will be used to define the RTW; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

In those cases were the standard deviation for a particular standard is zero (no difference between the 3 retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. The laboratory must calculate RTWs for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory and available for review.

6.2 Brief Description of Qualitative and Quantitative Analysis

If a peak is present within the RTW of a particular analyte on the primary column, the secondary column is evaluated to determine if the analyte indeed confirms and is within the RTW on the secondary column. The concentration of the analyte is calculated using peak area or height with respect to the average calibration factor from the initial calibration. If peak area is used to evaluate the initial calibration, then that parameter is used to verify continuing calibration verifications, as well as to quantitate sample results. Alternatively, peak height may be used for all.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 per batch [3]	<Reporting Limit (< ½ RL Project QAPP)
Lab Control Sample (LCS)	1 per batch [3]	Statistical Control Limits [4]
LCS Duplicate (LCSD) [1]	1 per batch [3]	Statistical Control Limits [4]
Matrix Spike (MS) [2]	1 per batch [3]	Statistical Control Limits [4]
MS Duplicate (MSD) [2]	1 per batch [3]	Statistical Control Limits [4]
Surrogate	every sample	Statistical Control Limits [4]

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project.
[2] The sample selection for MS/MSD, if not specified by the client on the chain-of-custody, is randomly selected by the extraction analyst within the preparation batch.
[3] A batch is not to exceed 20 samples.....pre-determined by the extraction department.
[4] Statistical control limits are established on a yearly basis and are subject to change (refer to Table 1).

Note: State specific or program specific acceptance criteria may be required, check the Project/Job notes in LIMs to determine if any custom limits are applicable. Ex. For the state of South Carolina, the LCS results must meet a 70 – 130% recovery window.

7.2 Sample Preservation and Storage

All samples should be collected in a glass container with a Teflon-lined lid. If the lid does not have a Teflon-liner, aluminum foil should be placed between the jar and lid (dull side facing the sample). All samples and extracts are stored at 4±2°C prior to analysis and must be analyzed within 40 days after extraction.

Matrix	Preservation [1]	Extraction Hold Time SW-846 (VTS) [2]	Analysis Hold Time
Soil/Sediment	Cool 4 ± 2°C	14 days	Within 40 days after Extraction
Water	Cool 4 ± 2°C	7 days	Within 40 days after Extraction

[1] Prior to and after extraction; and prior to analysis
[2] VTS = verified time of sampling

7.3 Sample Preparation

Refer to the extraction SOPs UP-SP-006 (Herbicide – Soils) and UP-SP-007 (Herbicide – Waters) for complete details.

7.4 Calibration / Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented in LIMS.

Calibration Controls	Sequence	Control Limit
Calibration Standards	6-pt. (min) linearity	≤20% RSD
Second Source (ICV/SSV)	Prior to samples	80 – 120% 85 – 115% (QAPP specific)
Cont. Cal. Verif. (CCV)	prior to and after every 10 sample injections	±20% pred. resp. ±15% pred. resp.(QAPP specific)
Retention Time Windows (RTW)	Initial CCV determines the midpoint of RTW	± 3x SD

7.4.1 External Standard Calibration

Each of the 6 concentration levels of the esterified standards are analyzed on the GC. One of the external standards should be at a concentration near, but above, the reporting limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

Inject each calibration standard using the technique that will be used to introduce the actual samples into the GC. Tabulate peak height or area response against the masses for each analyte. If the percent relative standard deviation (%RSD) of the calibration factor is <20% over the working range, linearity through the origin can be assumed, and the calibration factor (CF) for the middle level standard may be used in place of a calibration curve.

$$\text{Calibration Factor (CF)} = \frac{\text{total height of peak}}{\text{mass injected (nanograms)}}$$

The working calibration factor must be verified on each working day or at the beginning of every 72-hour sequence by injecting the middle calibration standard. ECDs that operate in the subnanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of the calibration is necessary. The CCV will be analyzed every 10 samples injections. If the response differs by more than ± 20%, a new calibration curve must be prepared for that analyte.

7.4.2 Calibration Evaluation and Acceptance Criteria

Refer to the TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (Attachment 2).

7.5 Preventive Maintenance

- The septa should be changed between each analysis sequence. The number of injections made plays a part in how frequently they should be changed. Generally, not more than 100-125 injections should be made without changing the septum.
- Clip about 1-meter from the guard column when later eluting compounds begin tailing.
- The entire system should be periodically checked including Hydrogen and Nitrogen tanks for leaks and loose wires.
- Leak tests must be performed every six months on every electron capture detector.
- Inspect injection end of column for particulates and/or cracks.
- Change connectors.
- Condition of the column - upper end of column temperature 285°C for 1 - 2 hours.
- Remove 1-6 inches from the injector and guard column (if present).
- Make sure septum doesn't need to be changed.
- Press connector should be checked after the instrument has been baked, if guard column is present

Refer to the instrument manuals if problems are encountered which can not be solved by routine maintenance.

7.6 Sample Analysis

7.6.1 Analytical Sequence

7.6.1.1 GC Conditions (May vary by instrument)

Function	Rate
Injector Temperature	Cool-on-column
Detector Temperature	300°C
Initial Column Temp.	120
Initial Hold	0.75 minutes
Ramp-1	20°C / minute
Final Temp / Hold-1	190°C / 0.0minute
Ramp-2	15°C / minute
Final Temp / Hold-2 Ramp-3 Final Temp / Hold-3	240°C / 0.0 minute 7.5 °C / minute 285 °C / 1.0 minutes

7.6.2 Gas Chromatographic Analysis

Because of the low concentration of the herbicide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column may need to be primed or deactivated by injecting the herbicide standard level 6 several times. These initial injections are performed prior to beginning initial or daily calibration.

Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed every 10 sample injections with mid-level CCV standards. The sequence must end with a mid-level CCV standard. The sequence must also stop when qualitative and/or quantitative QC criteria are exceeded.

7.6.3 Direct Injection

All calibration and samples are injected at 1.0 uL using the solvent flush technique via autosampler for better reproducibility. If sample compound(s) responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that the extract be diluted so that all of the peaks are on scale. Over-lapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms manipulated to ensure all peaks or on scale over a 100-fold range, is acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration. If peak detection is prevented by the presence of interferences, the sample may need to be diluted.

Calibrate the system immediately prior to conducting any analyses. This is accomplished by running the calibration standards. Standards must be injected at the intervals specified. The calibration factor for each analyte to be quantitated must not exceed a 20% difference when compared to the calibration standards. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with sample analysis. All samples that were injected after the sample exceeding the criteria must be re-injected.

Establish daily RTWs for each analyte. Use the absolute retention time for each analyte from the initial CCV as the midpoint of the window for the day. The daily RTW equals the midpoint +/- 3X the standard deviation.

Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily RTW. Normally, confirmation is required on a second GC column. It may be necessary to occasionally confirm something by GC/MS, but the concentration needs to be in the ppm levels before that is possible. Confirmation may not be necessary if the composition of the sample matrix is well established and the client agrees that no confirmation is necessary. This, however, is not the normal protocol.

If confirmation by GC/MS were required, the results would be flagged with a "C" and noted in the narrative what method was used for confirmation (i.e., SW846 Md. 8270C).

Validate the qualitative performance of the GC system by running the mid-level standards throughout the analysis sequence to evaluate this criterion. If any of the standards fall outside of their daily retention window, the system is out of control. Determine the cause of the problem and correct it.

7.6.4 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must identify, initial, and date the changes on the hardcopy report. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

- Manual integrations shall follow the TestAmerica Corporate SOP for manual integrations (CA-Q-S-002) located in Attachment 5. An example of one such integration and documentation is included in the attachment.

Manual integrations are most often performed for the following reasons.

Peak Not Integrated	Split Peak
Sample Matrix Interference	Assign Peak
Column Bleed	Baseline Smoothing
Instrument Noise	Isomers
Peak Tail	Shouldering
Incomplete Integration	Other
Wrong Peak	

A peer review is performed on all manual integrations. A pdf of all the raw data including the before and after manual integrations are automatically imported into the TALs AD batch from Chrom. A summary of the MIs are generated in the TALs deliverable and is include in the data summary portion of the data package. The pdf of the MIs are automatically included in the Level 4 deliverable

7.7 Documentation

7.7.1 Instrument Runlogs

The analysis of samples and standards is documented within each instrument-specific runlog (Attachment 3), and must be completed for each day's analysis.

7.7.2 Traceability of Standards

Upon receipt or preparation, each standard is entered into LIMS and is issued a unique ID# based upon the type and sequential order in which the item was received. Further information includes the manufacturer, lot # (if applicable), the date received or prepared, the expiration date, volume/weight received; concentration (if applicable); preparation details (if applicable), initials of the recording analyst, and the description of the item (i.e., **XXXX** Stock Solution – LCS/MS). Once the record is created, a unique label is printed and affixed to the appropriate standard/reagent container.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies and in accordance with the TestAmerica corporate procedure CA-T-P-003, *Reporting Results for Methods that Require Second-Column Confirmation* (Attachment 6). The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are

retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 At least one MB and LCS will be included in each laboratory lot of 20 or fewer samples. The MB will be examined to determine if contamination is being introduced in the laboratory. The MB and LCS must be carried through all stages of the sample preparation and measurement steps. The QA department tabulates these results and in-house control limits are generated.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Precision will be measured by the reproducibility of the MSs and will be calculated as Relative Percent Difference (RPD). If MSs were not analyzed, reproducibility will be measured using the LCS/LCD. Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 Surrogate compounds will be added to every analytical and QC sample to measure the performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.1.5 For each analytical batch (20 samples), an MB, LCS and MS/MSD must be analyzed. These samples must be carried through all stages of the sample preparation and the measurement steps. Also, every sample must be spiked with the surrogate DCAA. The QA department tabulates these results and in-house control limits are generated.

8.1.6 Each time an analytical sequence is started, the herbicide standard must be evaluated to determine if the chromatographic system is operating properly. The analyst should consider -- Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations for each compound? Careful examination of the standard chromatogram will indicate whether the column is still good, if the injector is leaking, or if the injector septum needs replacing, etc..

8.1.7 Required Instrument QC

8.1.7.1 The method requires that the grand mean %RSD vary by <20% when comparing calibration factors to determine if a 5-point minimum calibration curve is linear. Note there are state or program criteria that prohibit the use of the Grand Mean approach. Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the calibration curve. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed.

8.1.7.2 The method sets a limit of grand mean ± 20% difference when comparing the continuing response of a given analyte versus the initial response. Note there are state or program criteria that prohibit the use of the Grand Mean approach. Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the calibration verification standards. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed. If the limit is exceeded, corrective action must be taken to correct the problem or the sequence must be started over. All samples following the standard that was out must be reanalyzed.

8.1.7.3 The method requires the establishment of RTWs.

8.1.7.4 The method requires that all succeeding standards in an analysis sequence must fall within the daily RTW established by the first standard of the sequence.

8.2 Corrective Actions

Throughout Sections 6, 7 and 8, numerous criteria are described which must be met to meet analytical requirements. This section will go through what corrective actions should be taken by the analyst when any of the criteria are not met.

8.2.1 For external standard calibration it is required that a 5-point minimum linearity must be run initially. If the %RSD of the calibration factors for the linearity is >20%, corrective action must be taken. If this initial linearity cannot be met, first try re-running the standards. If the %RSD's are still unacceptable, perhaps new working standard solutions will need to be made.

8.2.2 The system must be calibrated immediately prior to analysis. This is performed as detailed in Section 6. If the % difference of the calibration factors exceed a 20% difference when compared to the initial linearities, corrective action must be taken. First try re-injecting the standards and if still out of control, then follow the actions described in Section 7. If this does not resolve the problem, new standard dilutions may be necessary.

8.2.3 If throughout the analytical sequence any CFs for any of the analytes exceeds the 20% difference limit, corrective action must take place. First, try re-injecting the standards. If that does not make a difference, refer to Section 7.0 for recommended maintenance. A good thing to remember: don't assume anything until it is proved to be within control limits! If the CF is still unacceptable, a new analytical sequence must be started. All samples run after the 20% difference criterion is exceeded and before the problem is solved will have to be re-analyzed.

8.2.4 If throughout the analytical sequence the analyst notices that the baseline becomes excessively noisy or that the sensitivity of the instrument drops or that the peaks start tailing excessively, refer to Section 7.0 or the instrument manual for corrective actions. Once the instrument has been returned to optimum operating conditions a new analytical sequence must be started. All samples run after a mid-level standard and before the problem was discovered will have to be re-analyzed.

8.2.5 When an out of control situation occurs, the analysts must use their best analytical judgment and available resources when determining the action to be taken. The out of control situation may or may not be caused by more than one problem. The analyst should seek the help of their immediate supervisor, QA personnel, or other experienced staff if they are uncertain of the cause of the out of control situation and the corrective action. The analysis must not be resumed until the source of the problem and an in control status is attained. All samples associated with the out of control situation must be reanalyzed. Out of control data must never be released without approval of the supervisor, project manager, QA personnel, or the lab manager. (A Non-Conformance Memo (NCM) must be written to document this 'approval'.)

Listed below are steps that must be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on a NCM;
- document in maintenance logbook that an in control situation has been achieved; and
- receive approval of the section manager, QA personnel, or the laboratory manager prior to release of any analytical data associated with the problem.

IF AT ANY TIME THE ANALYST IS UNCERTAIN AS TO WHAT ACTIONS TO TAKE OR HOW TO PERFORM SUGGESTED MAINTENANCE, PLEASE SEE YOUR SECTION MANAGER. IF SOMETHING UNUSUAL IS HAPPENING THAT IS NOT DESCRIBED IN THIS SECTION, SEE YOUR SECTION MANAGER. COSTLY (AS IN MONEY AND TIME) DAMAGE CAN RESULT TO THE INSTRUMENT, DETECTOR, OR COLUMN IF MAINTENANCE IS NOT PERFORMED CORRECTLY.

8.2.5.1 Calibration Factors

Correct the problem, and re-run the linearity and determine the new average calibration factor.

8.2.5.2 Initial Calibration Verification (ICV/SSV)

Repeat the standard to verify proper preparation. If still non-compliant, prepare a new standard from the original stock, recalibrate with a new standard curve, or prepare new stock and/or working standards.

8.2.5.3 Laboratory Control Sample (LCS)

The recoveries for the spiked compounds must be within the laboratory's internally-derived statistical control limits. Note: State specific or program specific acceptance criteria may be required, check the Project/Job notes in LIMs to determine if any custom limits are applicable. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze the LCS to determine if instrumental conditions or analytical preparation was the cause. If the LCS is still non-compliant and the samples are within the extraction holding time, then request re-extraction using a Non-Conformance Memo (NCM) and reanalyze all associated samples. If the samples are outside the extraction holding time, then contact the project manager using an NCM. Unless otherwise directed, samples will not be extracted outside the hold time and the data will be submitted with the appropriate narration.

8.2.5.4 LCS Duplicate (LCSD)

The LCSD must meet all control limits as LCS in addition to limits set for precision (same corrective action as LCS).

8.2.5.5 Method Blank (MB)

The MB should not contain any target compounds at or above the reporting limit. State or program specific criteria that the MB may not exceeding ½ the RL may also apply. If non-compliant, then the MB should be reanalyzed to determine if instrument contamination was the cause. If the MB is still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCM and reanalyze all associated samples containing target compounds at <10x amount found in the MB. If a sample contains target compounds at ≥ 10x amount found in the MB, or if target compounds are not detected in the sample, then that sample does not require re-extraction and the results may be reported without qualification. If the samples our outside the extraction holding time, then contact the project manager using an NCM. Unless directed otherwise, samples will not be extracted outside the holding time and the data will be submitted with appropriate narration.

8.2.5.6 Matrix Spike (MS)

The recoveries for the spiked compounds must be within the laboratory's internally-derived statistical control limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause.

8.2.5.7 Matrix Spike Duplicate (MSD)

See MS for recoveries. RPD's should be within the laboratory's internally-derived statistical control limits. If non-compliant, check calculations for documentable errors. Check unspiked sample results and surrogate recoveries for indications of matrix effects. If significant differences exist between the MS and MSD, reanalysis of the sample spikes may be necessary.

8.2.5.8 Surrogate Spike

The recoveries for the surrogate(s) must be within the laboratory's internally-derived statistical control limits. If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, and the surrogate recoveries in the MB and LCS are within control limits, then sample matrix effects are the most likely cause. However, any samples with surrogate recoveries significantly below the control limits with, no visible chromatographic cause should be reanalyzed to determine if an injection error was the cause for the low recovery. If the surrogate recoveries in the associated MB and LCS are not within control limits, and the samples are within the extraction holding time, then re-extract and reanalyze all associated samples. If the samples are outside the extraction holding time, then contact the project manager using and NCM. If the MS/MSD of the same sample is outside the control limits, this also confirms matrix interference. Unless otherwise directed, samples will not be re-extracted out of hold time and data will be submitted with appropriate narration.

8.2.5.9 Continuing Calibration Verification (CCV)

The continuing calibration standard corresponds to level 4 of the calibration curve (Attachment 1) The response factors of the initial calibration check standard should not vary from the response factors of the corresponding standard from the calibration curve by more than ± 20% on at least one column for each target compound, and on both columns for any compound reported positive. (See Section 8.1.7.1 CCVs can be a grand mean of ±20%) unless state or program criteria prohibit the use of the Grand Mean approach. Some state specific certification programs, including the state of South Carolina prohibit the use of the Grand Mean approach in evaluation of the calibration check standards. Refer to the analyst backlog and/or project/job notes to determine which state or program requirements need to be applied to the samples being analyzed. If any of the target compounds fail these criteria, then the standard is considered to be non-compliant. If the standard is non-compliant, then reanalyze the standard. If the standard is still non-compliant, remove 6-12 inches from the guard column change the connector and condition the column for 1-2 hours. If the standard is still non-compliant, then prepare a new standard. If the new standard is non-compliant, then recalibrate the instrument with a new curve. All target compounds which fail these criteria should be narrated as showing increased sensitivity, and how this might bias data. Notify the project manager of any non-compliance using the NCM. Unless directed otherwise, samples will not be analyzed outside of holding time and the data will be submitted with appropriate narration

8.2.5.10 Retention Time Windows

Evaluate the data for usability based on a comparison with the standards run during the analytical sequence. Also, consider the retention times for the surrogates and spiked compounds analyzed before and after the sample in question.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Concentration (ppb) $= \dfrac{[(A_1)(V_t)(Df)]}{[(V_i)(V_s)(D)]\,[CF]}$

Where:
A_1 = response for the analyte in the sample (area or height)
V_i = volume of extract injected (mL).
Df = dilution factor (if applicable)
V_t = volume of total extract (mL).
V_s = volume/weight of sample extracted
D = (100 - % Moisture) / 100
CF = Average calibration factor from the initial calibration curve

9.2 %RSD $= \dfrac{\text{Standard Deviation}}{\text{Mean}} \times 100$

Where,
Standard Deviation $= \left| \dfrac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1} \right|^{1/2}$

Where,
x_1 = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

9.3 % Difference $= \frac{\text{Avg. CF - CF}}{\text{Avg. CF}} \times 100$

Where:
Avg CF = Average CF from initial linearity
CF = CF from the analysis of the verification standard

9.4 Surrogate Percent Recovery $= \frac{Q_d}{Q_a} \times 100$

Where:
Q_d = Quantity determined by analysis
Q_a = Quantity added to sample/blank

9.5 Spike Recovery $= \frac{SR}{SA} \times 100$

Where:
SR = Spike result
SA = Spike added

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Flammable Vials" wastestream.
- Single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Example: Compound Listing and Reporting Limits

Attachment 1. Example: Initial Calibration Report and CCV Verification Report
Attachment 2. TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points'
Attachment 3. Example: Worklist Run Log Report / Maintenance Log
Attachment 4. Example: Data Review Checklist
Attachment 5. TestAmerica Corporate SOP, CA-Q-S-002, Acceptable Manual Integration Practices and example documentation
Attachment 6. TestAmerica Corporate Procedure, CA-T-P-003, Reporting Results for Methods that Require Second-Column Confirmation.

14.0 REVISION HISTORY

- Revision 17 updated on 7/31/13
- Annual Review
- Remove reference to the DoD QSM criteria
- Section 5.3.4 was updated to correct the working standard final volumes
- Section 5.3.1.2 was updated to reflect the current standard storage requirement.
- Sections 8.1.7.2 and 8.2.5.9 were updated to reflect current ICV/CCV criteria.
- Section 7.6.2 was updated to reflect the MI Reasons in Chrom

Table 1.

Example: Compound Listing and Reporting Limits
(023-001)

Note: Reporting limits will vary depending on sample size/volume, dry weight reporting for soils and dilution factors.

TestAmerica Chicago

Herbicide: 8151A_Reporting Limit Summary

Analytes	CAS #	RL	Units	Analyte Type
Water Matrix				
2,4,5-T	93-76-5	1.0	ug/L	Analyte/Parameter
2,4-D	94-75-7	1.0	ug/L	Analyte/Parameter
2,4-DB	94-82-6	1.0	ug/L	Analyte/Parameter
4-Nitrophenol	100-02-7	2.0	ug/L	Analyte/Parameter
Dalapon	75-99-0	2.0	ug/L	Analyte/Parameter
Dicamba	1918-00-9	1.0	ug/L	Analyte/Parameter
Dichlorprop	120-36-5	1.0	ug/L	Analyte/Parameter
Dinoseb	88-85-7	1.0	ug/L	Analyte/Parameter
Pentachlorophenol	87-86-5	0.5	ug/L	Analyte/Parameter
Picloram	1918-02-1	0.5	ug/L	Analyte/Parameter
Silvex (2,4,5-TP)	93-72-1	1.0	ug/L	Analyte/Parameter
2,4-Dichlorophenylacetic acid	19719-28-9		ug/L	Surrogate Cpnd
Solid Matrix				
2,4,5-T	93-76-5	33	ug/Kg	Analyte/Parameter
2,4-D	94-75-7	33	ug/Kg	Analyte/Parameter
2,4-DB	94-82-6	33	ug/Kg	Analyte/Parameter
4-Nitrophenol	100-02-7	NA	ug/Kg	Analyte/Parameter
Dalapon	75-99-0	66.7	ug/Kg	Analyte/Parameter
Dicamba	1918-00-9	33	ug/Kg	Analyte/Parameter
Dichlorprop	120-36-5	33	ug/Kg	Analyte/Parameter
Dinoseb	88-85-7	33	ug/Kg	Analyte/Parameter
Pentachlorophenol	87-86-5	16.7	ug/Kg	Analyte/Parameter
Picloram	1918-02-1	16.7	ug/Kg	Analyte/Parameter
Silvex (2,4,5-TP)	93-72-1	33	ug/Kg	Analyte/Parameter
2,4-Dichlorophenylacetic acid	19719-28-9		ug/Kg	Surrogate Cpnd
Waste Matrix				
2,4,5-T	93-76-5	1000	ug/Kg	Analyte/Parameter
2,4-D	94-75-7	1000	ug/Kg	Analyte/Parameter
2,4-DB	94-82-6	1000	ug/Kg	Analyte/Parameter
4-Nitrophenol	100-02-7	NA	ug/Kg	Analyte/Parameter
Dalapon	75-99-0	2000	ug/Kg	Analyte/Parameter
Dicamba	1918-00-9	1000	ug/Kg	Analyte/Parameter
Dichlorprop	120-36-5	1000	ug/Kg	Analyte/Parameter
Dinoseb	88-85-7	1000	ug/Kg	Analyte/Parameter
Pentachlorophenol	87-86-5	500	ug/Kg	Analyte/Parameter
Picloram	1918-02-1	500	ug/Kg	Analyte/Parameter
Silvex (2,4,5-TP)	93-72-1	1000	ug/Kg	Analyte/Parameter
2,4-Dichlorophenylacetic acid	19719-28-9		ug/Kg	Surrogate Cpnd
Leach Matrix				
2,4,5-T	93-76-5	100	ug/L	Analyte/Parameter
2,4-D	94-75-7	100	ug/L	Analyte/Parameter
2,4-DB	94-82-6	100	ug/L	Analyte/Parameter
4-Nitrophenol	100-02-7	200	ug/L	Analyte/Parameter
Dalapon	75-99-0	200	ug/L	Analyte/Parameter
Dicamba	1918-00-9	100	ug/L	Analyte/Parameter
Dichlorprop	120-36-5	100	ug/L	Analyte/Parameter
Dinoseb	88-85-7	100	ug/L	Analyte/Parameter
Pentachlorophenol	87-86-5	50	ug/L	Analyte/Parameter
Picloram	1918-02-1	50	ug/L	Analyte/Parameter
Silvex (2,4,5-TP)	93-72-1	100	ug/L	Analyte/Parameter
2,4-Dichlorophenylacetic acid	19719-28-9		ug/L	Surrogate Cpnd

(023-001)

Attachment 1.

Example: Initial Calibration Report and CCV Verification Report (024-001 to 024-003)

TestAmerica Laboratories
Initial Calibration Report

Method: \\Chi-svr07\ChromData\GC41-42\20130807-18240.b\inst41_42.m

Instrument: INST41-42 Lims Location: 500
Lock State: Unlocked Cpnd Order: Retention Time
Integrator: Genie Last Modified: 08-Aug-2013 12:56:55
No.Compounds:12

Initial Calibration Batches

Ical Batch: \\Chi-svr07\ChromData\GC41-42\20130807-18240.b
Inj Date : 07-Aug-2013 14:32:41, Sublist: chrom-inst41_42*sub1

Limit Group: GC_8151A_ICAL

Detector 1: Ch-A-01031141

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
1 Dalapon	Disabled	373010	297855	277103	272348	253406	14273	229564		Linr 0.999
2 4-Nitrophenol	749840	834790	673890	627813	598835	Disabled		697034		Avg 14
$ 3 2,4-Dichlorophenylacet	1984875	1973487	1601234	1487456	1359853	1319425		1621055		Avg 18
4 Dicamba	5615640	6091290	5118070	4855730	4530300	4443762		5109132		Avg 13
5 Dichlorprop	2066460	2172090	1825590	1700333	1583008	1542990		1815078		Avg 14
6 2,4-D	2427620	2360480	2541500	2263657	2050595	1988514		2272061		Avg 9.5
7 Pentachlorophenol	21886260	23367100	20228465	18996507	18014235	17466352		19993153		Avg 11
8 Silvex (2,4,5-TP)	9418940	10364820	8715870	8141967	7773898	7575972		8665244		Avg 12
9 2,4,5-T	9234720	10080400	8396435	7899780	7403898	7155806		8361840		Avg 13
10 2,4-DB	1789300	252870	1456170	1338307	1265090	1363360		207472		Avg 16
11 Picloram	1731760	12757820	1559855	1432260	1375000	1437522		1570705		Avg 13
12 Dinoseb	3934540	4212380	3575855	3366090	3374163	3356452		3636580		Avg 9.9

Detector 2: Ch-B-01031142

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
1 Dalapon	Disabled	1434560	1211040	1021953	1009758	927924	61544	839997		Linr 0.994
2 4-Nitrophenol	2795480	2896650	2416985	2208067	2081680	1939394		2389709		Avg 16
$ 3 2,4-Dichlorophenylacet	7322917	7267512	5941873	5292804	4936374	4538885		5883394		Avg 20
4 Dicamba	20160460	21812710	18525155	16901680	16113880	14982564		18082742		Avg 14
5 Dichlorprop	8505700	8790910	7327580	6468960	6141815	5626528		7143582		Avg 18
6 2,4-D	10037540	10384950	8725790	7726057	7375188	6895632		8524193		Avg 17
7 Pentachlorophenol	82910160	90868800	80118070	73740257	72106550	66829234		77762178		Avg 11
8 Silvex (2,4,5-TP)	37397800	41001330	36252395	33309990	32497278	30013700		35078749		Avg 11
9 2,4,5-T	38064220	40527620	35516280	32499190	31640823	29113048		34560197		Avg 12
10 2,4-DB	6830080	964420	6072150	5521680	5323198	4937120		804966		Avg 16
11 Picloram	7460420	55231340	6870595	6543903	6387400	6115276		6871414		Avg 9.7
12 Dinoseb	16487840	19283160	18030570	17110993	17221853	16565112		17449921		Avg 6.1

(024-001)

Report Date: 08-Aug-2013 14:48:59 Chrom Revision: 2.1 01-Aug-2013 12:45:24

Preliminary Report

TestAmerica Chicago
CCV, Cal Verification Report

Data File:	\\Chi-svr07\ChromData\GC41-42\20130807-18240.b\080513_136.D		
Lims ID:	CCV HERB	Client ID:	
Inject. Date:	07-Aug-2013 17:33:37	Dil. Factor:	1.0000
Sample Type:	CCV		
Sample ID:	#: dc= Name: 080513,herb41,500-0018240-010		
Misc. Info.:			
Operator:	WernerS	Instrument ID:	INST41-42
Injection Vol:	1.0 ul	ALS Bottle#:	0
Lims Batch ID:	18240	Lims Sample ID:	10
Sublist:	chrom-inst41_42*sub1		
Detector 1 :	Ch-A-01031141	Injection Date:	07-Aug-2013 17:33:37
Detector 2 :	Ch-B-01031142	Injection Date:	07-Aug-2013 17:56:23
Method:	\\Chi-svr07\ChromData\GC41-42\20130807-18240.b\inst41_42.m		
Last Update:	08-Aug-2013 14:45:42	Calib Date:	07-Aug-2013 16:26:10
Quant Method:	External Standard	Quant By:	Initial Calibration
Last ICal File:	\\Chi-svr07\ChromData\GC41-42\20130807-18240.b\080513_133.D		
Limit Group:	GC_8151A_ICAL		
Integrator:	Genie		
Column Type:	Rtx-CLP1	Column Dia:	0.53 mm
Process Host:	XAWRK030		
Start Cal Date:	07-Aug-2013 14:32:41		
End Cal Date:	07-Aug-2013 16:26:10		

Detector 1: Ch-A-01031141

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	%D	Max. %D
1 Dalapon	0.3000	-0.001	0.3044	280513	1.5	20
2 4-Nitrophenol	697034	-0.001		651377	-6.6	20
$ 3 2,4-Dichlorophenylaceti	1621055	0.001		1477213	-8.9	20
4 Dicamba	5109132	0.001		4802083	-6.0	20
5 Dichlorprop	1815078	0.001		1682087	-7.3	20
6 2,4-D	2272061	0.003		2171037	-4.4	20
7 Pentachlorophenol	19993153	-0.002		18799473	-6.0	20
8 Silvex (2,4,5-TP)	8665244	0.0		7990460	-7.8	20
9 2,4,5-T	8361840	0.0		7746997	-7.4	20
10 2,4-DB	207472	0.005		176807	-14.8	20
11 Picloram	1570705	0.007		1319883	-16.0	20
12 Dinoseb	3636580	-0.001		3181417	-12.5	20

Start Cal Date: 07-Aug-2013 14:32:41
End Cal Date: 07-Aug-2013 16:26:10

Detector 2: Ch-B-01031142

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	%D	Max. %D

(024-002)

Report Date: 08-Aug-2013 14:48:59 Chrom Revision: 2.1 01-Aug-2013 12:45:24

Preliminary Report

Data File: \\Chi-svr07\ChromData\GC41-42\20130807-18240.b\080513_136.D

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	%D	Max. %D
1 Dalapon	0.3000	0.0	0.3138	1083740	4.6	20
2 4-Nitrophenol	2389709	0.0		2398290	0.4	20
$ 3 2,4-Dichlorophenylaceti	5883394	0.0		5285135	-10.2	20
4 Dicamba	18082742	0.001		16706860	-7.6	20
5 Dichlorprop	7143582	0.0		6314083	-11.6	20
6 2,4-D	8524193	0.001		7521953	-11.8	20
7 Pentachlorophenol	77762178	0.0		69792657	-10.2	20
8 Silvex (2,4,5-TP)	35078749	0.001		31856290	-9.2	20
9 2,4,5-T	34560197	0.001		31355670	-9.3	20
10 2,4-DB	804966	0.001		707950	-12.1	20
11 Picloram	6871414	0.001		6253420	-9.0	20
12 Dinoseb	17449921	0.0		15277183	-12.5	20

(024-003)

Attachment 2.

TestAmerica Corporate Procedure, CA-T-P-002, 'Selection of Calibration Points' (025-001 to 025-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

06/18/2012		12/21/2012	
Technical Services Director	Date	Quality and EH&S Director	Date

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(025-001)

1.0 PURPOSE

This policy describes TestAmerica Laboratories, Inc. requirements for the determination of the number of points and removal of points from calibration curves.

2.0 SCOPE

This policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP), then that requirement will be used for the defined procedure.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the DQOs for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a single point at the reporting level.

Examples:

A. Required to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by HPLC-UV at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the DQOs will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in the laboratory for meeting a regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A 5-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where the technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS's used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the QA Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of points from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24-hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internal standards show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (All analytes in spike mix must show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection.)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers), the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.
- Revision 2.1, dated 11 June 2012.
  - General clarifications – no procedural changes.

Attachment 3.

Example: Worklist Run Log Report / Maintenance Log (026-001 to 026-003)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 080713 ICAL Worklist Num: 18240
Instrument: INST41-42 Method: inst41_42
Batch Directory: \\Chi-svr07\ChromData\GC41-42\20130807-18240.b
Anaylysis Type: SemiVOA Creator: Werner, Sharon A
Inj Volume: 1.00 Inj Vol Units: ul

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
PRIME	500-0018240-001	Client		07-Aug-2013 14:10:18	080513_127.D		1.0		sv
STD6 HERB	500-0018240-002	IC	6	07-Aug-2013 14:32:41	080513_128.D		1.0		sv
STD5 HERB	500-0018240-003	IC	5	07-Aug-2013 14:55:27	080513_129.D		1.0		sv
STD4 HERB	500-0018240-004	ICRT	4	07-Aug-2013 15:18:02	080513_130.D		1.0		sv
STD3 HERB	500-0018240-005	IC	3	07-Aug-2013 15:40:42	080513_131.D		1.0		sv
STD2 HERB	500-0018240-006	IC	2	07-Aug-2013 16:03:31	080513_132.D		1.0		sv
STD1 HERB	500-0018240-007	IC	1	07-Aug-2013 16:26:10	080513_133.D		1.0		sv
ICV	500-0018240-008	ICV		07-Aug-2013 16:48:52	080513_134.D		1.0		sv
ICV	500-0018240-009	ICV		07-Aug-2013 17:11:02	080513_135.D		1.0		sv
CCV HERB	500-0018240-010	CCV		07-Aug-2013 17:33:37	080513_136.D		1.0		sv
MB 500-195923/1-A	500-0018240-011	MB		07-Aug-2013 17:56:23	080513_137.D		1.0		sv
LCS 500-195923/2-A	500-0018240-012	LCS		07-Aug-2013 18:18:43	080513_138.D		1.0		sv
500-58834-A-35-C	500-0018240-013	Client		07-Aug-2013 18:41:21	080513_139.D		1.0	WP Herbicides	sv
500-58834-A-35-C	500-0018240-014	Client		07-Aug-2013 19:04:12	080513_140.D		5.0	WP Herbicides	sv
500-58834-A-35-C	500-0018240-015	Client		07-Aug-2013 19:26:53	080513_141.D		10.0	WP Herbicides	sv
HEXANE	500-0018240-016	Client		07-Aug-2013 19:49:24	080513_142.D		1.0		sv
CCV HERB	500-0018240-017	CCV		07-Aug-2013 20:11:52	080513_143.D		1.0		sv
MB 500-194304/1-A	500-0018240-018	MB		07-Aug-2013 20:34:17	080513_144.D		1.0		sv
LCS 500-194304/2-A	500-0018240-019	LCS		07-Aug-2013 20:56:37	080513_145.D		1.0		sv
LCSD 500-194304/3-A	500-0018240-020	LCSD		07-Aug-2013 21:18:59	080513_146.D		1.0		sv
500-58834-A-29-A	500-0018240-021	Client		07-Aug-2013 21:41:21	080513_147.D		10.0	WP Herbicides 4np	sv
500-58834-A-29-A	500-0018240-022	Client		07-Aug-2013 22:03:40	080513_148.D		50.0	WP Herbicides 4np	sv
HEXANE	500-0018240-023	Client		07-Aug-2013 22:26:10	080513_149.D		1.0		sv
CCV HERB	500-0018240-024	CCV		07-Aug-2013 22:48:36	080513_150.D		1.0		sv
MB 500-196700/1-A	500-0018240-025	MB		07-Aug-2013 23:11:07	080513_151.D		1.0		sv
LCS 500-196700/2-A	500-0018240-026	LCS		07-Aug-2013 23:33:31	080513_152.D		1.0		sv
LB 500-196503/1-D	500-0018240-027	LB		07-Aug-2013 23:56:43	080513_153.D		1.0		sv
LB 500-196506/2-B	500-0018240-028	LB		08-Aug-2013 00:19:02	080513_154.D		1.0		sv
310-12663-A-1-K	500-0018240-029	Client		08-Aug-2013 00:41:18	080513_155.D		1.0	3072524-01	sv
310-12663-A-1-L MS	500-0018240-030	MS		08-Aug-2013 01:03:39	080513_156.D		1.0		sv
500-60279-A-1-F	500-0018240-031	Client		08-Aug-2013 01:26:07	080513_157.D		1.0	JL-SG09-045W-ST01	sv
500-60279-A-2-E	500-0018240-032	Client		08-Aug-2013 01:48:27	080513_158.D		1.0	JL-JLM132-ST01-07	sv
HEXANE	500-0018240-033	Client		08-Aug-2013 02:10:46	080513_159.D		1.0		sv
CCV HERB	500-0018240-034	CCV		08-Aug-2013 02:33:04	080513_160.D		1.0		sv
MB 500-196906/1-A	500-0018240-035	MB		08-Aug-2013 02:55:21	080513_161.D		1.0		sv
LCS 500-196906/2-A	500-0018240-036	LCS		08-Aug-2013 03:17:40	080513_162.D		1.0		sv
LB3 500-196666/1-D	500-0018240-037	LB3		08-Aug-2013 03:40:01	080513_163.D		1.0		sv
490-31737-A-1-C	500-0018240-038	Client		08-Aug-2013 04:02:15	080513_164.D		1.0	CWS Radio Tower D	sv

(026-001)

Worklist Run Log: 080713 ICAL

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
490-31737-A-1-D MS	500-0018240-039	MS		08-Aug-2013 04:24:29	080513_165.D		1.0		sv
HEXANE	500-0018240-040	Client		08-Aug-2013 04:46:46	080513_166.D		1.0		sv
CCV HERB	500-0018240-041	CCV		08-Aug-2013 05:09:11	080513_167.D		1.0		sv
CCV HERB	500-0018240-042	CCV		08-Aug-2013 10:19:19	080513_169.D		1.0		sv
ICV	500-0018240-043	ICV		08-Aug-2013 10:41:44	080513_170.D		1.0		sv
ICV	500-0018240-044	ICV		08-Aug-2013 11:03:59	080513_171.D		1.0		sv
CCV HERB	500-0018240-045	CCV		08-Aug-2013 11:26:26	080513_172.D		1.0		sv

(026-002)

TestAmerica Chicago
GC Extractables Instrument Maintenance Log

Page No.:_____
Instrument No.: 41 / 42

Routine/Informal Maintenance includes: Glass Y-connectors checked for leaks; periodic system check for leaks, frayed wires and a general overview; flows checked

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Entry No.:_____
Date of Maintenance:_____ Analyst:_____

___ Septa Changed (~100 injections)
___ Heated the Column/Detector for Cleaning
___ Injection Liner Changed
___ Y-connectors changed
___ Column Changed / Clipped (circle one)
___ O-rings Replaced
___ Other (explain)

Major Power Outage Occurred: _____

Explain/Actions Taken:_____

Demonstration of Return to Control:
___ CCV analyzed and in-control.
___ Samples Reanalyzed.
___ Other (explain)

Explain:_____

Reviewer Signature:_____ Date:_____

Attachment 4.

Example: Data Review Checklist
(027-001 to 027-003)

GC/HPLC Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
REPORTS:		
p-Flag report reviewed and accepted?		
Retention Time Study reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: <20% RSD; for 608 and 610 < 10% RSD		
Linear or Quadratic Regression curve fit for all >20% RSD R^2 >0.990		
Plots for all Linear Regressions reviewed?		
conc-intercept < RL; (<1/2 RL QAPP specific); <30% Error (Chrom: Details of Calibration per Analyte)		
Co-eluting peaks reviewed and accepted?		
All manual integrations reviewed found to be necessary, performed correctly and have documented reasons?		
Method:		
Method locked in Chrom?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct and locked in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 85-115% (15%D) criteria? 8015C; 8082A; 8081B (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date_____

Secondary Reviewer: _____ Date_____

(027-001)

TestAmerica Chicago
GC EXTRACTABLES / HPLC DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Analytical Method: a) GC _____ b) HPLC _____ Instrument (Primary / Confirmation):_____

Extraction Method: a) SW 3510 b) SW 3541 c) SW 3550 d) SW 3580 e) Other (_____)

Matrix: WATER / SOIL / SPLP-TCLP / Waste / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: _____ _____ _____ _____ _____
Import Batch Review: Is the Calibration ID Correct? Y N Report Status of Primary / Secondary correct? Y N CCV's are correct and properly linked to samples? Y N Calculation of all QC complete? Y N			
View All Data Review: *Samples:* Results reported for all samples? Y N Dilutions / Re-analyses are correctly represented? Y N NA Are flags correctly assigned? Y N *QA Summary: QC Association Summary* All required dilutions/re-analyses present? Y N All Samples linked to prep batch? Y N All Samples linked to batch QC? Y N *Lab QC:* All QC present and correctly represented? Y N *Surrogate*: All samples/QC present and correct? Y N			
Form 1 / View All Data: IF **original** and **re-analyses** are to be reported in LIMS: Suffix Used: Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Sample # Original Dilution Comments
FORM 2 / View All Data: Surrogate Recoveries within Limits? Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3 / View All Data: MS/MSD Recoveries Acceptable Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3 / View All Data: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits: _____ QAPP: _____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

(027-002)

TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Criteria Met QAPP specific MB < ½ RL _____ NCM Ref Number: _____			
FORM 4: 1) Primary and Secondary Column info present? Yes No NA 2) All Samples Present Yes No NA 3) Sulfur and GPC clean-ups identified? Yes No NA 4) Header Information Correct? Yes No NA NCM Ref Number: _____			
FORM 8: Retention Time Window Criteria Met? Yes No NCM Ref Number: _____			
Form 10: 1) Retention Time Window Criteria Met? Yes No NA 2) All hits present/Concentrations Correct? Yes No NA 3) Are hits < 40%D between primary & secondary columns? Yes No (NCM Ref No.: _____)			
RAW DATA: 1) Raw Data Verified and Complete? Yes No NA			
2) Raw Data Matches Forms? Yes No NA			
3) Sample info complete-% solids, Smp.Wt, F.V.?			
4) Prep/Extraction Data info verified in Raw Data?			
Form 6: Initial Calibration Criteria met and Complete? Samples linked to ICAL? _____			
Form 7: CCV criteria met method criteria Samples linked to CCV? _____ NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Blank / LCS / LCSD Yes No 2) MS/MSD Yes No NA 3) MRL Check Required: Yes No Limits Applied: _____			
Manual Integration reports (before and afters) present and reasons correctly documented and approved?			
Prep / Sample Summary pages Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required / Properly Documented? Yes No			
Manual Calculation of On Column result: Peak Height or Area of Analyte Average Peak Height or Area from ICAL			Sample : _____ Compound: _____
JOB STATUS: 1) Primary Reviewer/Analyst: 1st / 2nd Level			
2) Secondary Reviewer/Supervisor: Lab Complete			
Additional Comments:			

Attachment 5.

TestAmerica Corporate SOP, CA-Q-S-002, 'Acceptable Manual Integration Practices' (028-001 to 028-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

Raymond J. Frederici 08/01/2012
Raymond J. Frederici — Date
Quality and EH&S Director

Larry Penfold 08/01/2012
Larry Penfold — Date
Quality Compliance Director

Verl D. Preston 08/01/2012
Verl D. Preston — Date
Quality Assessment Director

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(028-001)

1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

4.5 **Baseline:** The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 **Peak:** An increase in signal from baseline to a maximum and then back to baseline.

4.7 **Coelution:** The concurrent elution of two or more compounds to the detector at the same time.

4.8 **Elution:** The process of movement of compounds from the chromatographic system.

4.9 **Carry-over:** Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 **Peak Tailing:** Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 **Interference Peaks or Fused Peaks:** Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important considerations in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters is necessary to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibrations standards, QC standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. This

process for before & after chromatograms is also required for OVAP-approved laboratories, unless instructed otherwise.

5.4.7.1 Re-integration technique marked, if available, on the data system.

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.

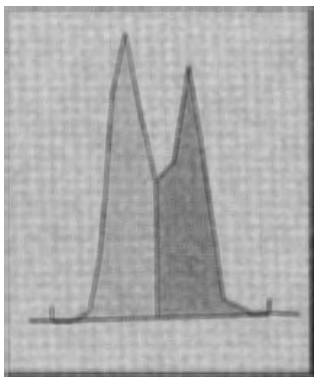
- Expanded section on data miner software to include Chrom Audit Miner
- Included reference to data miner training presentation.

- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples

Attachment 1.

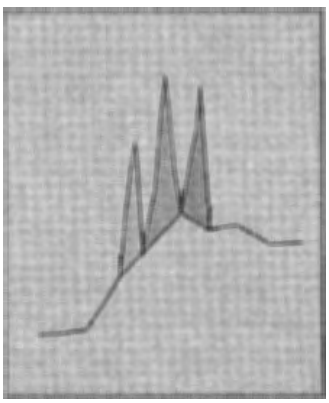
Examples of Peak Shape and Proper Integration Documenting Manual Integrations

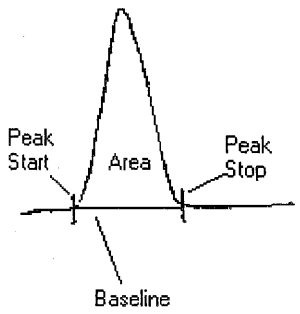
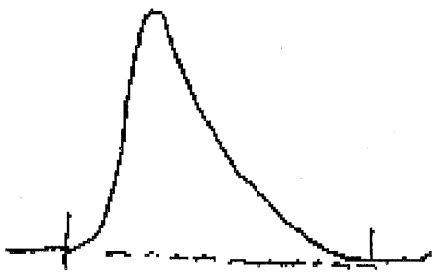
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.

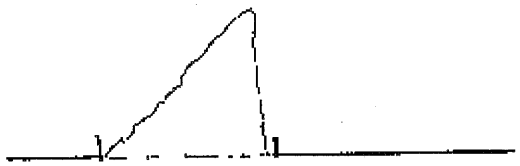
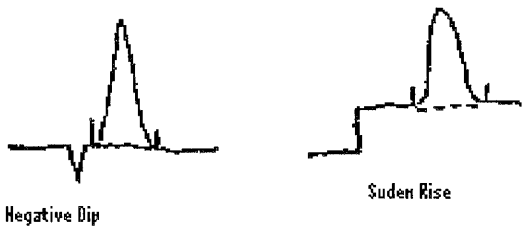


Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.

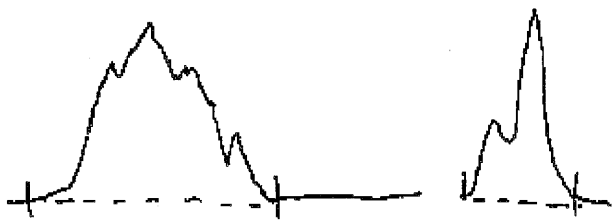
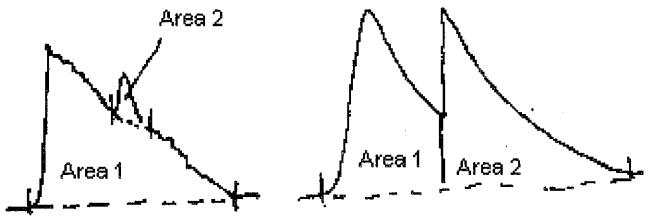
Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.



Ideal peak (Gaussian Shape) The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	

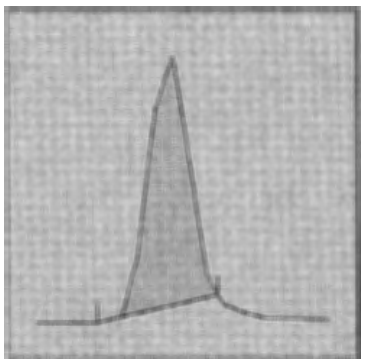
(028-012)

Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

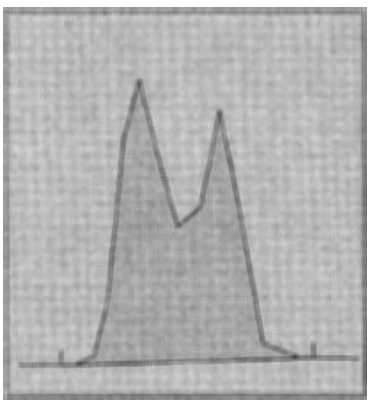
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

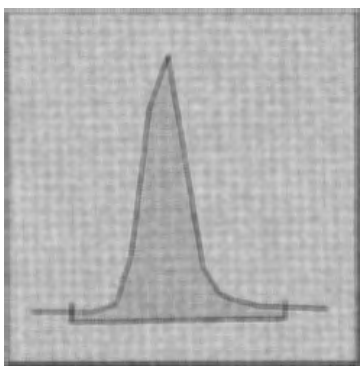
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



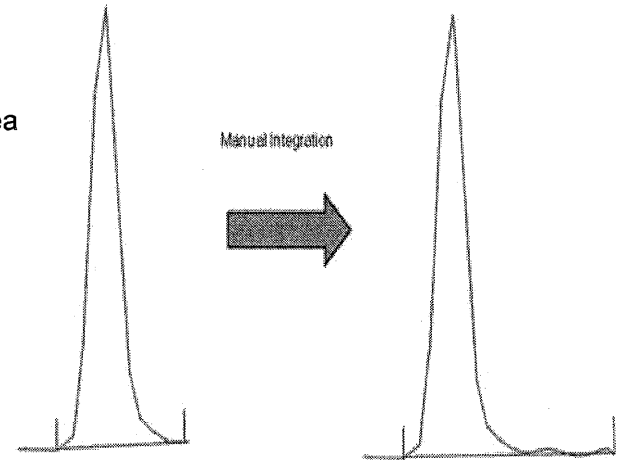
Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.



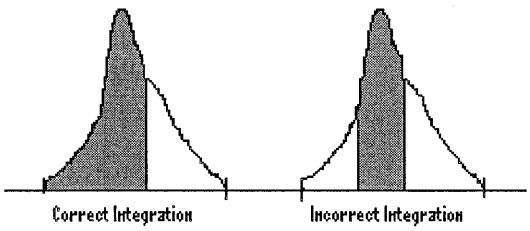
Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 6.

TestAmerica Corporate Procedure, CA-T-P-003
Reporting Results for Methods that Require Second-Column Confirmation
(029-001 to 029-003)

Reporting Results for Methods that Require Second-Column Confirmation

Approvals (Signature/Date):

Technical Services Director	06/18/2012 Date	Quality and EH&S Director	12/21/2012 Date

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(029-001)

1.0 PURPOSE

This policy describes a standardized approach to reporting results for methods that require second-column confirmation, such as EPA SW-846 8000 series GC methods. This policy is designed to be in compliance with EPA methods 8000B and 8000C as well as EPA 600 series methods and is intended to be followed in the absence of QAPP or regulatory guidance to the contrary (e.g., DOD, DOE, and CLP).

2.0 SCOPE

This policy applies to all methods that require second-column (qualitative and quantitative) confirmations. This procedure is not applicable to qualitative only confirmations, such as Methods 8330 or 8330A, which report only from the primary column.

3.0 SAFETY

Refer to Laboratory-specific method SOPs.

4.0 DEFINITIONS

None.

5.0 PROCEDURE

5.1 The laboratory may designate a primary column for each analyte (in which case the second column is the confirmation column for each analyte), or may report the lower result from the two columns. The same column does not have to be primary for every analyte. Both columns are intended to be calibrated and meet calibration verification requirements.

5.2 Whichever approach is used, QC must be assessed and reported in the same way as the samples (i.e., report from the primary column or report the lower number).

5.3 40% RPD Criterion

5.3.1 When using the primary column option, if the RPD for the dual column results is $\leq$ 40%, then the result from the primary column is normally reported. The exception is situations where a chromatographic interferent is evident in the primary column chromatogram that is not present in the confirmation column chromatogram.

5.3.2 If using the primary column option and the RPD for the dual column results is > 40%, then the lower value is reported. (A RPD of >40% is clear evidence of chromatographic interference on the higher value). A case narrative comment or footnote discussing the RPD failure must be included in the final report.

5.4 Calibration Verification Criterion – Primary Column Option

5.4.1 If the CCV recovery for the primary column is within acceptance limits and the analyte is confirmed on the secondary column, but the secondary column is outside acceptance limits then results from the primary column may be reported with a case narrative comment or footnote.

5.4.2 If the CCV recovery for the confirmation column is within acceptance limits and the recovery for the primary column is in the range of +15% to +40%, then results may be reported from the confirmation column with a case narrative comment or footnote.

5.4.3 If the CCV recovery is below acceptance limits for the primary column, or the secondary column is below acceptance limits and the analyte is not confirmed, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.4.4 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

5.5 Calibration Verification Criterion – Lower Result Option

5.5.1 If the CCV recovery for the column with the lower result is within acceptance limits and the analyte is confirmed on the secondary column but the secondary column is outside acceptance limits, then results from the lower column may be reported with a case narrative or footnote.

5.5.2 If the CCV recovery for the higher result is within acceptance limits but the lower result column fails acceptance limits and the RPD is < 15%, then the higher result may be reported with a case narrative or footnote.

5.5.3 If the CCV recovery on either column is more than 40% above the acceptance limit, then the problem should be investigated and fixed. The associated samples will need to be reanalyzed under a new successful calibration.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving second-column confirmation are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 6 November 2007
  - Initial Release.
- Revision 1, dated 23 June 2009
  - No Changes
- Revision 2, dated 18 June 2012
  - Added quantitative column confirmation
  - Added limits for CCV failures on confirmation columns; and clarified language.

TITLE: Gas Chromatography / Mass Spectrometry - Semi-Volatiles SW-846 Method 8270D

Approvals (Signature/Date):

Gary L. Rynkar — Date 11/28/12 Supervisor, GC/MS BNA	Mark Storm — Date 11/28/12 Env.Health & Safety Coordinator
Terese A. Preston — Date 11/28/12 Quality Assurance Manager	Michael J. Healy — Date 11/29/12 Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the analysis of semi-volatile base/neutral and acid (BNA) compounds by Gas Chromatography/Mass Spectrometry (GC/MS). This SOP was written using SW-846 4th Edition, Methods 8270D and 8000 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative and/or Non-Conformance Memo (NCM).

Specific requirements pertaining to the DOD QSM Version 4.2 are located in Appendix A. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are ~3-5x the respective MDL to ensure confidence in the value reported.

Reporting limits [a.k.a., Practical Quantitation Limits (PQLs)] are assessed annually based on MDL performance. If the MDL does not meet the routine laboratory reporting limit or the method specified limit, it is repeated, or the laboratory reporting limit is reassessed. Table 2 defines the laboratory's reporting limits for each compound that can be routinely analyzed by this method according to each matrix.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Methods

This method is appropriate for the analysis of BNA's in wastewaters, soils, sediments, solids, sludges, wastes, and oils. Concentrated extracts are transferred to crimp-top vials, internal standard solution added, with subsequent analysis on the GC/MS. The method finds applicability for most BNA compounds that are soluble in dichloromethane (DCM, a.k.a., Methylene chloride) and chromatograph well on a fused-silica capillary column. Table 1 lists those compounds with their characteristic ions. The approximate retention times were generated by a specific GC/MS system.

The following exceptions are noted: Benzidine is subject to loss during solvent concentration and chromatographs poorly. Hexachlorocyclopentadiene is subject to decomposition in the inlet, chemical reaction in acetone and photochemical decomposition. N-nitrosodimethylamine can be difficult to separate from the solvent front. N-nitrosodiphenylamine decomposes in the injection port and cannot be separated from diphenylamine. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

Those samples suspected of high background levels may be screened by a GC/MS prior to analysis. Initial dilutions are determined from this screen.

Instrument calibration occurs prior to analysis and every 12 hours of run-time. Instrument maintenance is performed as needed, on a daily, monthly or yearly basis, as the specific action requires.

2.0 INTERFERENCES

External interference's can be caused by contaminants from sample containers, preparative glassware and reagents, inlet liners, syringes and columns and manifest themselves in discrete peaks and/or elevated baselines. "Soap" peaks and phthalates are the most common external interferences encountered. Proper glassware preparation, sample handling, and instrument maintenance should reduce the contamination from these sources. A laboratory reagent blank is analyzed with each batch to demonstrate the absence of these contaminants.

Rinsing the syringes well, both the syringe used for sample prep and sample injection, prior to and after use, for each sample or standard is critical. Assigning separate functions to the syringes prevents cross-contamination (introduction of compounds from one sample to a sample not originally containing those compounds). Replacing the rinse and DCM dilution containers often is also necessary.

Carryover is another source of contamination. After analysis of highly concentrated samples or background containing samples, a DCM blank should be run, or at a minimum, the oven temperature should be raised to 300°C and baked out.

Internal or matrix interference's can be co-extracted from the sample during preparation. These can appear as large distinct peaks and/or elevated baselines. Occasionally, matrix interferences manifest themselves as low/high recoveries of spike compounds. Matrix interferences vary from sample to sample. Unless specifically requested by the client, clean-up procedures are not performed to remove heavy background or interferences.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, non-absorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The GC/MS contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the GC and the MS. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride (DCM)	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

4.1 GC/MSD

A temperature-programmable GC is equipped with a splitless injection port and accessories (syringes, gases, and columns). The column used is a DB5MS, 30m x 0.25mm ID or equivalent bonded-phase silicone coated fused-silica capillary column, with 0.50um film thickness. Interfaced with a mass-selective detector capable of scanning from 35 to 500 amu every second or less using 70 volts electron energy in the Electron Ionization (EI) mode. The system must be able to produce an acceptable mass spectrum of decafluorotriphenylphosphate (DFTPP) when 50-ng is injected on the column.

4.2 Data System

The analytical system is interfaced with a data system capable of continuing acquisition and storage of mass spectral data. The software allows plotting of specific masses versus time or scan numbers (Extracted Ion Current Profile - EICP) and integration of that abundance. The system also contains the latest NBS Library.

4.3 Accessories

- 10 uL, 25 uL, 500 uL, 1000 uL and 2500 uL gas-tight syringes.
- Amber and clear vials (1.5 mL and 4.5 mL)
- Teflon/silicone septa
- Crimp-top and screw-top vial caps

4.4 Current Hardware/Software

- 1 – 5890 Series II Plus Gas Chromatographs with Electronic Pressure Control (EPC), Hewlett-Packard (HP)
- 4 – 6890N Gas Chromatograph with Electronic Pressure Control (EPC), Agilent
- 2 – 7890A Gas Chromatograph with Electronic Pressure Control (EPC), Agilent
- 3 – 5975 Mass Selective Detector, Agilent
- 4 – 5973 Mass Selective Detectors, Agilent
- 2 – 7693 Autosampler and Accessories, Agilent
- 1 – 7683B Autosampler and Accessories, Agilent
- 4 – 7683 Autosampler and Accessories, Agilent
- 1 – 7673 Autosampler and Accessories, Hewlett-Packard
- 7 – Agilent ChemStations B.02.04 software and peripheral hardware
- 1 – Agilent Chemserver with 3.5.1
- 1 – Chrom data system Version 1.2

5.0 REAGENTS AND STANDARDS

All neat standards or stock solutions received are entered into the laboratory's TALS. A standard label is printed from TALS and affixed to the bottle or ampule. All standards are then stored at -10°C to -20°C until needed. All glassware and syringes needed to prepare reagents and standards must be cleaned, dried and rinsed well with the solvent to be used (methanol and DCM).

All information concerning standard preparation must be entered into TALS. The labels contain the following information: Standard ID, solution description (content, weights, concentrations, date prepared, expiration date and analyst initials). All neat standards (referenced by standard type and date in TALS) used in preparations are noted and cross-referenced here, providing a standard

tracking mechanism. The expiration date of any prepared standard cannot exceed the expiration date assigned for any neat standard that is a component in the prepared standard mixture.

5.1 Surrogate Parent Spiking Solution

The surrogate spiking solution is obtained from Cerilliant as a prepared solution in a 100 mL amber glass Teflon-lined screw-capped bottle. The surrogate spike is prepared by Cerilliant using EPA certified standards at the following concentrations:

Compound	Concentration (ug/mL)
p-Terphenyl-d_{14}	1000
Nitrobenzene-d_5	1000
2-Fluorobiphenyl	1000
Phenol-d_5	1500
2-Fluorophenol	1500
2,4,6-Tribromophenol	1500

- Life of Standard: 12-months from date of transfer of standard or less as indicated by the manufacturers expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.1.1 Surrogate Working Spike Solution

5 mLs of surrogate stock + 95 mLs MeOH in a 100 mL amber glass Teflon-lined screw-capped bottle. The working surrogate spike is prepared by the extractions department at the following concentrations:

Compound	Concentration (ug/mL)
p-Terphenyl-d_{14}	50
Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
Phenol-d_5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75

- Life of Standard: 12-months from date of transfer of standard or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

The surrogate spike solution is spiked in all samples, the matrix spike/matrix spike duplicate (MS/MSD) samples, method blanks (MB), and the laboratory control sample (LCS) at a volume of 0.5 mLs prior to extraction. This results in a spiking level of 37.5 ug for the Acid surrogate compounds and 25 ug for the Base/Neutral surrogate compounds.

5.1.2 Surrogate Working Spike Solution

5 mLs of surrogate stock + 95 mLs MeOH in a 100 mL amber glass Teflon-lined screw-capped bottle. The working surrogate spike is prepared by the extractions department at the following concentrations:

Compound	Concentration (ug/mL)
p-Terphenyl-d_{14}	50
Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
Phenol-d_5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75

- Life of Standard: 12-months from date of transfer of standard or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

The surrogate spike solution is spiked in all samples, the matrix spike/matrix spike duplicate (MS/MSD) samples, method blanks (MB), and the laboratory control sample (LCS) at a volume of 0.5 mLs prior to extraction. This results in a spiking level of 37.5 ug for the Acid surrogate compounds and 25 ug for the Base/Neutral surrogate compounds.

5.2 LCS and Matrix Spike Parent Solution

The full list spike solution is prepared by the extractions department from stock solutions. The parent spiking solutions are prepared from stock solutions from Cerilliant using EPA certified standards at the following concentrations:

5.2.1 8270 LCS Mix Parent

The 8270 LCS Mix Calibration Stock is purchased from Cerilliant at a 200 ug/mL concentration with 5 mLs volume.

Acenaphthene	1,2-Dichlorobenzene	2-methylphenol (o-Cresol)
Acenaphthylene	1,3-Dichlorobenzene	4-methylphenol (p-Cresol)
Acetophenone	1,4-Dichlorobenzene	Naphthalene
Aniline	3,3-Dichlorobenzidine	2-Nitroaniline
Anthracene	2,4-Dichlorophenol	3-Nitroaniline
Azobenzene	2,6-Dichlorophenol	4-Nitroaniline
Benzo(a)anthracene	Diethylphthalate	Nitrobenzene
Benzidine	2,4-Dimethylphenol	2-Nitrophenol
Benzo(b)fluoranthene	Dimethylphthalate	4-Nitrophenol
Benzo(k)fluoranthene	Di-n-butylphthalate	N-Nitroso-di-n-butylamine
Benzo(g,h,i)perylene	2,4-Dinitrophenol	N-Nitrosodiethylamine
Benzo(a)pyrene	2,4-Dinitrotoluene	N-Nitrosodimethylamine
Benzoic acid	2,6-Dinitrotoluene	N-Nitroso-di-n-propylamine
Benzyl alcohol	Dinoseb	N-Nitrosodiphenylamine
Biphenyl	Di-n-octylphthalate	N-Nitrosomethylethylamine
bis(2-Chloroethoxy)methane	1,4-Dioxane	N-Nitrosopiperidine
bis(2-Chloroethyl)ether	Diphenyl ether	N-Nitrosopyrrolidine
Bis(2-Chloroisopropyl)ether	2-Ethoxyethanol	Pentachlorobenzene
bis(2-Ethylhexyl)phthalate	Fluoranthene	Pentachloroethane
4-Bromophenylphenylether	Fluorene	Pentachlorophenol
Butylbenzylphthalate	Hexachlorobenzene	Phenanthrene
Carbazole	Hexachloro-1,2-butadiene	Phenol
p-Chloroaniline	Hexachlorocylcopentadiene	Pyrene
4-Chloro-3-methylphenol	Hexachloroethane	Pyridine
2-Chloronaphthalene	Hexachlorophene	1,2,4,5-Tetrachlorobenzene
2-Chlorophenol	Hexachloropropene	2,3,4,6-Tetrachlorophenol
4-Chlorophenylphenylether	Indeno(1,2,3-c,d)pyrene	1,2,4-Trichlorobenzene
Chrysene	Isophorone	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	4,6-Dinitro-2-methylphenol	2,4,6-Trichlorophenol
Dibenzofuran	2-Methylnaphthalene	

- Life of Standard: 12-months or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.2.2 8270 LCS 3,3'-Dimethylbenzidine Parent

The 8270 LCS 3,3'-Dimethylbenzidine Parent Stock is purchased from Cerilliant at a 2,000 ug/mL concentration with 1 mL volume.

3,3'-Dimethylbenzidine

- Life of Standard: 12-months or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.2.3 LCS and MS Working Spike Solution

50 mLs of 8270 LCS Mix Parent Stock + 5 mLs of 8270 LCS 3,3'-Dimethylbenzidine Parent Stock are brought to a 100 mL final volume with DCM. These results in a spiking solution of 8270 HSL compounds at 50 ug/mL. This is stored in a 100 mL labeled amber, screw top vial with Teflon lined septum, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 6-months from date of preparation or less as indicated by the manufacturer.

The spiking solution is spiked in all LCS and MS/MSD samples at a volume of 1.0 mL prior to extraction. This results in a spiking level of 50 ug/mL for all compounds.

5.2.4 Initial Calibration (IcalSpike) Spike Solution

The ICV (Initial Calibration Spike) solution's are prepared from a stock solution purchased from Supelco (100 ug/mL) and Restek (2000 ug/mL) to result in a spike level of 50 ug/mL, for most compounds. Only the routinely analyzed 8270D compounds are present in the spikes. See attachment 9 for list of compounds and example recoveries for both mixes.

5.3 DFTPP / DDT / Pentachlorophenol (PCP) / Benzidine Stock Solutions

The DFTPP/DDT stock is purchased from Supelco at 1000 ug/mL; the PCP stock is purchased from Supelco at 5000 ug/mL; and the Benzidine stock is purchased through Supelco at 1000 ug/mL. All stocks are transferred to amber-glass, screw-topped, Teflon lined vials; assigned a TALS ID Number; and documented in TALS. Stock standards are stored at -10° to -20°C. An expiration date is assigned for 1-year from date of transfer or less as indicated by the manufacturer.

5.3.1 DFTPP / DDT / PCP / Benzidine Parent Solution

250 uLs of DFTPP/DDT stock + 50 uLs of PCP stock + 500 uL of Benzidine stock are brought to a 1000 uL final volume with DCM. This results in a parent solution of DFTPP/DDT/PCP at 250 ug/mL and Benzidine at 500 ug/mL. This is stored in a 1.5 mL labeled amber, screw top; vial with Teflon lined septum, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 12 months from date of preparation or less as indicated by the manufacturer.

5.3.2 The DFTPP / DDT / PCP / Benzidine Working Solution

100 uLs of the DFTPP/DDT/PCP/Benzidine Parent Solution are added to 900 uLs of DCM, resulting in a concentration of 50 ng/uL DFTPP/DDT/PCP and 100 ng/uL Benzidine. This is stored in a 1.5 mL labeled amber, crimp-top, autosampler vial, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 12-months from date of preparation or less as indicated by the manufacturer.

5.4 Internal Standard Solution

This solution is obtained as a mixture from Cerilliant:

Internal Standards (4,000 ug/mL)	
1,4-Dichlorobenzene- d_4	Phenanthrene- d_{10}
Naphthalene- d_8	Chrysene-d_{12}
Acenaphthene- d_{10}	Perylene- d_{12}

Once the 1.0 mL ampule is opened, the entire contents are transferred to a 1.5-mL amber gas-tight conical vial and labeled appropriately. All preparation procedures are recorded in TALS. Unopened ampules can be kept for the period indicated by the manufacturer, and are stored at -10° to -20°C until needed.

Refer to Table 5 for a listing of Internal Standards and corresponding ions assigned for quantitation.

- Life of Standard: 12-months or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.4.1 Internal Standard Working Solution

600 uLs of the Internal Standard Solution are added to 2400 uLs of DCM, resulting in a concentration of 800 ng/uL of Internal Standard. The entire contents are kept in a 3.0-mL amber gas-tight conical vial and labeled appropriately. All preparation procedures are recorded in TALS. The vials are stored at -10° to -20°C until needed.

- Life of Standard: 6-months or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.5 Initial Calibration Standards

Two separate initial calibrations are prepared; one for HSL and one for Appendix IX.

5.5.1 HSL Calibration Stock

The HSL Calibration Stock is purchased from Cerilliant at a 200 ug/mL concentration (except where noted) with a 5.0 mL volume.

Acenaphthene	Diethylphthalate	2-Nitroaniline
Acenaphthylene	p-Dimethylaminoazobenzene	3-Nitroaniline
Acetophenone	7,12-	4-Nitroaniline
2-Acetylaminofluorene	Dimethylbenz(a)anthracene	Nitrobenzene
4-Aminobiphenyl	3,3-Dimethylbenzidine	2-Nitrophenol
Aniline	2,4-Dimethylphenol	4-Nitrophenol
Anthracene	Dimethylphthalate	N-Nitroso-di-n-butylamine
Azobenzene	1,3-Dinitrobenzene	N-Nitrosodiethylamine
Benzo(a)anthracene	2,4-Dinitrophenol	N-Nitrosodimethylamine
Benzidine	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
Benzo(b)fluoranthene	2,6-Dinitrotoluene	N-Nitrosomethylethylamine
Benzo(k)fluoranthene	Dinoseb	N-Nitrosopiperidine
Benzo(g,h,i)perylene	Di-n-octyl phthalate	N-Nitrosopyrrolidine
Benzo(a)pyrene	Diphenylamine	5-Nitro-o-toluidine
Benzyl alcohol	Ethylmethanesulfonate	Pentachlorobenzene
bis(2-Chloroethoxy)methane	Fluoranthene	Pentachloroethane
bis(2-Chloroethyl)ether	Fluorene	Pentachloronitrobenzene
bis(2-Chloroisopropyl)ether	Hexachlorobenzene	Pentachlorophenol
bis(2-ethylhexyl)phthalate	Hexachloro-1,3-butadiene	Phenacetin
4-Bromophenylphenylether	Hexachlorocylcopentadiene	Phenanthrene
Butylbenzylphthalate	Hexachloroethane	Phenol
Carbazole	Hexachlorophene	2-Picoline
p-Chloroaniline	Hexachloropropene	Pronamide
Chlorobenzilate	Indeno(1,2,3-c,d)pyrene	Pyrene
4-Chloro-3-methylphenol	Isodrin	Pyridine
2-Chloronaphthalene	Isophorone	Safrole
2-Chlorophenol	Isosafrole	1,2,4,5-Tetrachlorobenzene
4-Chlorophenylphenylether	Kepone	2,3,4,6-Tetrachlorophenol
Chrysene	2-Methylcholanthrene	o-Toluidine
Diallate	4,6-Dinitro-2-methylphenol	1,2,4-Trichlorobenzene
Dibenz(a,h)anthracene	Methylmethanesulfonate	2,4,5-Trichlorophenol
Dibenzofuran	2-Methylnaphthalene	2,4,6-Trichlorophenol
Di-n-butylphthalate	2-methylphenol (o-Cresol)	1,3,5-Trinitrobenzene
1,2-Dichlorobenzene	3-methylphenol (m-Cresol)	**Surrogates:**
1,3-Dichlorobenzene	4-methylphenol (p-Cresol)	2-Fluorobiphenyl
1,4-Dichlorobenzene	Naphthalene	2-Fluorophenol (320 mg/mL)
3,3-Dichlorobenzidine	1,4-Naphthoquinone	Nitrobenzene-d5
2,4-Dichlorophenol	1-Naphthylamine	Phenol-d5 (320 mg/mL)
2,6-Dichlorophenol	2-Naphthylamine	p-Terphenyl-d14
		2,4,6-Tribromophenol (320 mg/mL)

- Life of Standard: 12-months or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.5.2 Surrogate Standard Solution - Initial Calibration Stock

The surrogate standard solution is obtained from Cerilliant as a prepared solution in a 1.0 mL amber vial. The surrogate standard is prepared standards at the following concentrations:

Compound	Concentration (ug/mL)
p-Terphenyl-d_{14}	4000
Nitrobenzene-d_5	4000
2-Fluorobiphenyl	4000
Phenol-d_5	4000
2-Fluorophenol	4000
2,4,6-Tribromophenol	4000

- Life of Standard: 12-months from date of transfer of standard or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.5.2.1 Surrogate – Calibration Parent Solution

50 uLs of the additional compounds stock standard is added to 950 uLs of DCM, resulting in a concentration of 200 ng/uL for the surrogates:

Compound	Concentration (ug/mL)
p-Terphenyl-d_{14}	200
Nitrobenzene-d_5	200
2-Fluorobiphenyl	200
Phenol-d_5	200
2-Fluorophenol	200
2,4,6-Tribromophenol	200
2-Chlorophenol-d4	200
1,2-Dichlorobenzene-d4	200

- Life of Standard: 12-months from date of transfer of standard or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store at -10 to -20°C. All bottles are labeled with a TALS ID Number.

5.5.3 Calibration Standard – Working (20 ug/L)

100 uLs of the 8270 BNA Working Calibration Standard Stock (200 ug/L) + 100 uL of surrogate Calibration Parent Solution (200 ug/L) is brought to a 1000 uL volume with DCM, resulting in a concentration of 20 ug/L. This is stored in a 1.5-mL labeled amber, Teflon-top vial, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 12-months from date of preparation or less as indicated by the manufacturer.

5.5.3.1 Calibration Standard – Working (5 ug/L)

250 uLs of the Calibration Standard - Working (20 ug/L) is brought to a 1000 uL volume with DCM, resulting in a concentration of 5 ug/L. This is stored in a 1.5-mL labeled amber, Teflon-top vial, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 12-months from date of preparation or less as indicated by the manufacturer.

5.5.4 HSL Working Initial Calibration Standards

Remove the HSL Calibration Stock and parent surrogate solution as identified in Section 5.5.1 and 5.5.2.1 from storage. Allow the standards to come to room temperature. Transfer the stock standards to 1.5-mL amber-glass, Teflon-lined, screw-capped vials. Record the transfer in TALS and obtain a TALS ID Number for each standard. Store the unused standard at -10° to -20°C.

Locate and rinse WELL with DCM, a 10 uL, 25 uL, 100 uL, 500 uL, and 1000 uL syringe.

The preparation information for the Working HSL Standards is entered into TALS and each standard is given a TALS ID Number. Prepare the Working Standards as follows:

Calibration Std.	BNA Working Std. (200 ug/mL)	Surrogate Parent (200 ug/mL)	DCM	Internal Std. (800 ug/mL)
80 ng/uL	250 uL	200 uL	Dilute to 0.5 mLs	*5uL
70 ng/uL	218.75 uL	175 uL	Dilute to 0.5 mLs	*5uL
50 ng/uL	156.25 uL	125 uL	Dilute to 0.5 mLs	*5 uL
40 ng/uL	125 uL	100 uL	Dilute to 0.5 mLs	*5 uL
20 ng/uL	62.5 uL	50 uL	Dilute to 0.5 mLs	*5 uL
10 ng/uL	31.25 uL	25 uL	Dilute to 0.5 mLs	*5 uL
5 ng/uL	15.625 uL	12.5 uL	Dilute to 0.5 mLs	*5 uL
2 ng/uL	200 uL of 5 ug/mL Parent Soln.	5 uL	Dilute to 0.5 mLs	*5 uL
1 ng/uL	100 uL of 5 ug/mL Parent Soln.	2.5 uL	Dilute to 0.5 mLs	*5 uL
0.5 ng/uL	50 uL of 5 ug/mL Parent Soln.	---	Dilute to 0.5 mLs	*5 uL
0.1 ng/uL	10 uL of 5 ug/mL Parent Soln.	---	Dilute to 0.5 mLs	*5 uL

* Add internal standard **after** the standard has been brought up to 0.5 mL.

- Life of Standard: 12-months from date prepared or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store in an amber crimp-top autosampler vials at -10 to -20°C.

5.6 Appendix IX Initial Calibration Stock Standards

The Appendix IX Supplemental Mix 2 Stock Standard, the EPA 8270/Appendix IX Semivolatile Stock Standard, Benzidines Mix, the Aramite Stock Standard, the Hexachlorophene Stock Standard, and the p-Phenylenediamine Stock Standard are purchased from Supelco in 1.0 mL sealed amber-glass ampules. The standards are labeled with a TALS ID Number and are stored at -10° to -20°C and can be kept for 1-year from the date of receipt or less as indicated by the manufacturer.

Each mixture contains the following compounds and concentrations:

8270 APIX Supplemental Mix 2 at 2,000 ug/mL	
Acetophenone	N-Nitrosodiethylamine
Isosafrole	N-Nitroso-di-n-butylamine
Pentachlorobenzene	N-Nitrosomethylethylamine
Phenacetin	2-Picoline
Methyl methanesulfonate	Safrole
N-Nitrosomorpholine	Hexachloropropene
N-Nitrosopiperidine	p-(Dimethylamino)azobenzene
N-Nitrosopyrrolidine	Ethyl methanesulfonate
Pentachloroethane	Pentachloronitrobenzene
o-Toluidine	3-Methylcholanthrene
1,2,4,5-Tetrachlorobenzene	1,3,5-Trinitrobenzene
1,3-Dinitrobenzene	1-Naphthylamine
2-Naphthylamine	2,3,4,6-Tetrachlorophenol
2,6-Dichlorophenol	2-Acetylaminofluorene
Dinoseb	4-Aminobiphenyl
4-Nitroquinoline-1-oxide	5-Nitro-o-toluidine
7,12-Dimethylbenz(a)anthracene	Diphenylamine

8270 Supplemental Mix 2 at 2,000 ug/mL	
1,4-Dioxane	1,4-Naphthoquinone
1,4-Dinitrobenzene	1-Methylnaphthalene
a,a-Dimethylphenethylamine	Chlorobenzilate
Dibenzo(a,j)acridine	2,3,5,6-Tetrachlorophenol
Diallate	Isodrin
Kepone	Pronamide

Single Component Stock Standards	
Compound	**Concentration (ug/mL)**
Aramite	2,000
Hexachlorophene	5,000
p-Phenylenediamine	5,000

5.7.2 Appendix IX Parent Solution - 200 ug/mL

Stock Standard	Concentration (ug/mL)	Amt. Added (uL)
8270 Calibration Mix 1	1,000	200
8270 Supplemental Mix 2	2,000	100
Aramite	2,000	100
p-Phenylenediamene	5,000	100
Hexachlorophene	5,000	320

The Appendix IX Parent Solution is brought up to a 1.0-mL final volume with DCM. The preparation is entered into TALS and the standard is given a TALS ID Number. An expiration date of 12-months is assigned or less as indicated by the manufacturer and the standard is stored in a 1-mL amber-glass, screw-capped vial with a Teflon septum at -10 to -20°C.

5.7.2.1 Appendix IX Parent Solution – (20 ug/mL)

50 uLs of the Appendix IX Parent Solution (200 ug/L) is brought to a 500 uL volume with DCM, resulting in a concentration of 20 ng/uL. This is stored in a 1.5-mL labeled amber, Teflon-top vial, at -10 to -20°C until needed. All preparation information is recorded in TALS and assigned a TALS ID Number. An expiration date is assigned for 12-months from date of preparation or less as indicated by the manufacturer.

5.7.3 Appendix IX Initial Calibration Standards

Remove the Appendix IX stock standards as identified in Section 5.6 from storage. Allow the standards to come to room temperature. Transfer the Appendix IX stock standards to 1.5 mL amber-glass, Teflon-lined, screw-capped vials. Enter the transfer information into TALS and obtain ID Numbers for each standard. Store the Appendix IX stock standards at -10°C to -20°C.

Locate and rinse WELL with DCM, a 10 uL, 25 uL, 100 uL, 500 uL, and 1000 uL syringe.

The preparation information for the Working Initial Calibration Appendix IX Standards is entered into TALS and each standard is given a TALS ID Number. Prepare the Working Standards as follows:

Calibration Std.	App. IX Parent Std. (200 ug/mL)	DCM	Internal Std. (800 ug/mL)
80 ng/uL	200 uL	Dilute to 0.5 mLs	*5 uL
70 ng/uL	175 uL	Dilute to 0.5 mLs	*5 uL
60 ng/uL	150 uL	Dilute to 0.5 mLs	*5 uL
50 ng/uL	125 uL	Dilute to 0.5 mLs	*5 uL
40 ng/uL	100 uL	Dilute to 0.5 mLs	*5 uL
20 ng/uL	50 uL	Dilute to 0.5 mLs	*5 uL
10 ng/uL	25 uL	Dilute to 0.5 mLs	*5 uL
5 ng/uL	12.5 uL	Dilute to 0.5 mLs	*5 uL

*Internal Standard added after each standard is brought to a 0.5 mL final volume with DCM.

The calibration levels listed above are correct for all compounds except the following for **Hexachlorophene:**

640
600
560
400
320
160
80
40

- Life of Standard: 12-months from date prepared or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store in amber crimp-top autosampler vials at -10 to -20°C.

5.8 Calibration Verification Standards

5.8.1 HSL Calibration Verification Standard

Remove the 8270 BNA Calibration Standard Stock (200 ug/mL), the DFTPP/DDT (1000 ug/mL) Stock Standard, and the Surrogate Parent (200 ug/mL) standard from storage. Allow the standards to come to room temperature.
Locate and rinse WELL with DCM, a 10 uL, 25 uL, 100 uL, 500 uL, and 1000 uL syringe.

The preparation information for the HSL calibration verification check standard is entered into TALS and each standard is given a TALS ID Number. Prepare the HSL calibration verification check standard weekly as follows:

Cal. Std.	BNA Calibration Mix (200 ug/mL)	Working DFTPP/DDT Mix (optional -1000ug/mL)	Surrogate Parent (200 ug/mL)	DCM	Internal Std. (800 ug/mL)
40 ng/uL	100uL	25uL	100 uL	Bring to 0.5 mL	*5 uL

* Add internal standard **after** the standard has been brought up to 1.0 mL.

- Life of Standard: 12-months from date prepared or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store in an amber crimp-top autosampler vials at -10 to -20°C.

5.8.2 Appendix IX Calibration Verification Standard

The Appendix IX calibration verification standard is prepared from the stock standards listed in Section 5.5.2. Bring the stock standards from storage and allow them to come to room temperature.

Locate and rinse WELL with DCM, a 10 uL, 25 uL, 100 uL, 500 uL, and 1000 uL syringe.
The preparation information for the Appendix IX calibration verification check standard is entered into TALS and the standard is given a TALS ID Number. Prepare the Appendix IX calibration verification check standard as follows:

Calibration Std.	Appendix IX Parent Std. (200 ug/mL)	DCM	Internal Std. (800 ug/mL)
40 ng/uL	100 uL	Brought to 0.5 mL	*5 uL

* Internal Standard is added after the standard is brought up to 0.5 mL.

The calibration levels listed above are correct for all compounds except for the following:

Compound	Calibration Std.
Hexachlorophene	320 ng /uL

- Life of Standard: 12-months from date prepared or less as indicated by the manufacturer's expiration date.
- Storage Requirements: Store in an amber crimp-top autosampler vials at -10 to -20°C.

6.0 CALIBRATION

Before any instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 Initial Calibration

An initial calibration is completed:

- as needed - calibration verification cannot be met;
- after a source cleaning and/or column change; and
- anytime a major repair or change has occurred with the instrument that affects calibration.

Confirm that the GC/MSD is stable and equilibrated. If possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/ changed.

Prior to beginning initial calibration:

- check the interface nut for security;
- check the vacuum (at 40°C - its normally between 4 and 5 x 10^{-5} torr);
- check the background, air/water levels and base ion by scanning for appropriate ions (44,32,28,18) and visually inspecting the spectrum scan [base ion is normally 28 (nitrogen) and good background levels are below 2000]; and
- check the stability of inlet helium flow generated by the Electronic Pressure Control (EPC).

6.2 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas (Perfluorotributylamine-PFTBA) are monitored at a temperature of 40°C and a column flow of 1.0 mL/minute. Monitoring at this temperature and flow produces the most representative cal gas scan, and therefore the best estimate of DFTPP response.

6.2.1 Set oven temperature to 40°C.

6.2.2 If an AUTOTUNE is to be performed, continue below. If not, continue to Section 6.2.5. An autotune is generally performed when the instrument has been down for maintenance (i.e., source cleaning or other non-routine maintenance).

6.2.3 After running an autotune, note the following:

6.2.3.1 Observe peak shape, absence of fronting, tailing, and the resolution between isotopes and mass axis for 69, 219 and 502 in the profile scan. The following is a guideline for the ratios of 69, 219, and 502, which indicate the source is operating properly.

Autotune report	
Isotope	**% of 69**
69	100 %
219	> 40 %
502	> 1 %

6.2.3.2 If the %'s do not meet these limits, the DFTPP standard will most likely not meet the required criteria. In addition, this is probably an indication that the source is not operating properly or perhaps the source is assembled incorrectly or it is dirty. Repeat the Autotune process and re-evaluate. If it still does not meet these limits, refer to the MSD manual for troubleshooting suggestions.

6.2.4 If the AUTOTUNE results are consistent with the limits above, go into MANUAL TUNE and save the AUTOTUNE values into the DFTPP tune file. The DFTPP tune file is used by the methods in setting the tune values (i.e. entrance lens, ion focus, etc..) to the values that were set in MANUAL TUNE. Once in MANUAL TUNE, adjust the tune parameters to meet the following limits:

Manual Tune Report	
Isotope	**% of 69**
69	100%
219	30-50%
502	1-2.5%

If unable to achieve these %'s redo an AUTOTUNE, and if still unsuccessful, refer to the MSD manual for troubleshooting suggestions.

6.2.5 If an AUTOTUNE has NOT just been performed, perform a MANUAL TUNE and adjust any parameters, if need be, to meet the relative abundances listed above. Adjustment may not be necessary, and not desired, if no previous problems have been encountered in meeting tune and calibration verification criteria. Hardcopy the profile scan and store the tune in the appropriate tune file.

6.3 DFTPP Analysis

Once the instrument is tuned, a 50 ng/uL injection of DFTPP must meet certain criteria. 1uL of the working DFTPP/DDT/PCP/Benzidine standard, containing 50 ng/uL of DFTPP/DDT/PCP and 100 ng/uL of Benzidine is analyzed by the method in Attachment 1.

6.3.1 The manner of acquiring the mass spectrum of DFTPP is performed in this way with Chrom software:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is performed and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. This procedure of averaging and subtracting is performed automatically by the Chrom software and evaluated by the criteria in Table 3.

6.3.2 The abundance's of designated masses MUST meet the criteria in Table 3, prior to any further analysis.

6.3.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%.

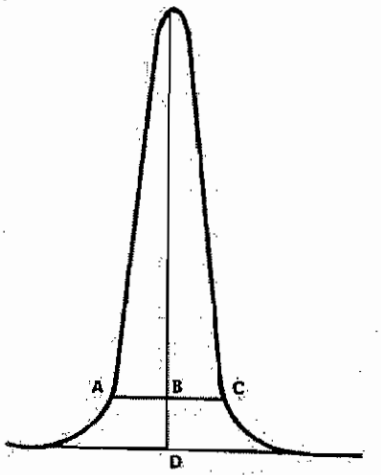
DDT Breakdown calculation

$$\text{\% breakdown of 4,4'-DDT} = \frac{\text{total DDT degradation peak area (DDE + DDD)}}{\text{total DDT peak area (DDT + DDE + DDD)}} \times 100$$

Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2, given by the following equation:

Tailing Factor = BC/AB

Where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height.



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

6.4 Analysis of Initial Calibration Standards

The HSL initial calibration and the Appendix IX initial calibration are usually analyzed on separate 12-hour tunes. Each calibration standard is analyzed according to the method in Attachment 1. The method outlined in Attachment 1 is an example and minor adjustments often have to be made to the method in regards to scan start/ending time. The electron multiplier (EM) voltage should remain constant corresponding to the voltage of the DFTPP analysis. Characteristic ions and approximate retention times of the compound are listed in Table 1. Initial calibrations are generally performed on an "as needed basis" (i.e., calibration standard cannot be met) or after major maintenance (i.e., source cleaning, column change) have been performed.

6.4.1 Analyze an initial calibration.

6.4.1.1 Quantitate each standard and update the calibration into the appropriate method.

6.4.1.2 Print an Initial Calibration (Attachment 3). Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each analyte using the following equations. The RSD should be less than or equal to 20% for each analyte (15% for DOD). It is also recommended that a minimum response factor for the most common analytes, as noted in Table 4, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also strive to place more emphasis on meeting the calibration criteria for those compounds that are critical project compounds, rather than meeting the criteria for those less important compounds. The data system calculates all RF's and %RSD and the calculation is as follows:

$$RF = \frac{A_t}{A_i} \times \frac{C_i}{C_t}$$

Where:

A_t= Area of compound
A_c= Area of internal standard
C_i = Concentration of internal standard
C_t = Concentration of compound

Each time a new HSL initial calibration is required, the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis.

$$\text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i = RF for each of the calibration standards

$\overline{RF}$ = mean RF for each compound from the initial calibration

n = Number of calibration standards e.g., 5

6.4.1.3 Application of Regression Curves in the Initial Calibrations (8270D)

For all compounds in the initial calibration with a %RSD >20.0%, calibration curves of area ratio versus concentration using a first or higher order regression curve of the calibration curve points will be performed. A first order linear regression or weighted linear regression will be used if it gives the highest correlation coefficient and if the y-intercept is > 0. Otherwise, a second or third order regression curve will be used. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient 0.995 (R^2=0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform instrument maintenance, then repeat the calibration procedure.

Method 8000/8270D specifies a minimum coefficient of determination of 0.995 (R^2=0.99). The methods also specify a minimum of 5 calibration points for a weighted linear (first order) model and a quadratic (second order) model requires six points. The laboratory, in order to meet AFCEE/DOD requirements, will analyze a minimum number of points to satisfy both SW-846 and AFCEE/DOD.

The recipes noted above will be modified to include the necessary calibration levels. Recipes are for guidance only and may change as needed.

Note: South Carolina does not allow the use of quadratic curves for initial calibrations. If the %RSD criteria are not met, only the linear regression model will be used. This will be considered a project-state-specific requirement.

6.4.2 Linearity of analytes -- If the RSD of any analyte is 20% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

6.4.2.1 If the RSD of any analyte is greater than 20%, then either the weighted linear or quadratic option can be used. No more than 10% of the compounds can fail the 20% RSD/0.99 correlation requirement. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

6.4.2.2 When the RSD exceeds 20%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

6.4.2.3 The average RF should not be used for compounds that have an RSD greater than 20% unless the concentration is reported as estimated. Our current computer system is unable to automatically flag these occurrences, only a manual flag is available. In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit. The procedure for adequate sensitivity is as follows: a standard at or below ½ the RL is analyzed at the same time as the initial calibration, the analyte in question is detected in this standard without need for manual integration then the analyte may be reported.

6.4.2.4 The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration

calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

6.4.3 Refer to the TestAmerica Corporate Procedure, CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points (Attachment 2).

6.4.4 The HSL initial calibration is verified via a second-source standard (see section 5.2.4), following the initial calibration and prior to the analysis of samples. The value determined from the second source check should be within 30% of the expected concentration. An alternative recovery limit within 20% is required for DOD QSM 4.2. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results could be used for screening purposes and would be considered estimated values. Corrective actions for out-of-control compounds in the ICV are assessed on a case-by case basis.

6.5 Calibration Verification

A calibration verification occurs every 12 hours of run-time. All DFTPP requirements must be met. After having satisfied these requirements, a daily calibration verification must be met.

6.5.1 Analyze 1 uL of the HSL calibration verification check standard according to the method specified in Attachment 1. If the Appendix IX compounds specified in Section 5.5.2 are to be analyzed, then the Appendix IX calibration verification standard will also be analyzed on the same 12-hour tune according to the method specified in Attachment 1. The method outlined in Attachment 1 is an example and minor adjustments often have to be made to the method in regards to scan start/ending time. The electron multiplier (EM) voltage should remain constant according to the voltage of the DFTPP analysis.

6.5.1.1 Each of the most common analytes in the calibration verification standard should meet the minimum response factors as noted in Table **4**. These criteria are particularly important when the common analytes are also critical project-required compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Possible problems include 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.

6.5.1.2 All compounds of interest must be evaluated using a 20% criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

$$\% \text{ Drift} = \frac{C_i - C_c}{C_i} \times 100$$

Where:
C_i = CCC standard concentration (50 ng)
C_c = measured CCC concentration

6.5.1.3 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 numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may 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 when the failed compound is present, the concentrations must be reported as estimated values.

6.5.1.4 Acceptable demonstrations of adequate sensitivity include any one of the following:

A. The calibration verification fails on the high side
B. A low, level continuing calibration verification standard (LLCCV) at or below the quantitation limit (RL) is analyzed in the same 12 hour batch as the sample in question, and the analyte in question is detected without need for manual integration. There is no recovery limit for this standard, the only requirement is detection.
C. A standard at or below ½ the RL is analyzed at the same time as the initial calibration, the analyte in question is detected in this standard without need for manual integration and the calibration verification %D is better than -50%.

6.5.1.5 All internal standard retention times in the calibration verification must not have changed more than 30 seconds from the midpoint standard in the HSL initial calibration. All internal standard areas must not change by a factor of 2 (-50% to +100%) from the midpoint standard in the HSL initial calibration.

6.5.1.6 The resolution between the closely eluting isomers benzo (b) fluoranthene and benzo (k) fluoranthene is checked to see if the height of the valley between the two peaks is less than 50% of the average of the two peak heights.

6.5.2 If both the DFTPP and calibration verification standard have met the required criteria, analysis can proceed.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Control is accomplished through:

1) daily tuning and calibration checks and
2) preparation QC traceable through individual extraction batches.

NOTE: Table 2 contains the laboratory internally-derived statistical control limits for the MS/MSDs, the LCS, and the surrogates. The limits used to assess precision and accuracy as well as the number of compounds being used for bench level control may vary with client, QAP, project, etc.. This information is transmitted to the bench via the COC, kick-off meetings, tech profiles, etc..

7.1.1 Initial Calibration

PFTBA
DFTPP.....Prior to initial calibration
Initial Cal. Stds.....Prior to samples
ICV (IcalSpike).....Prior to samples

7.1.2 Daily Analysis

PFTBA
DFTPP.....Prior to calibration verification
Daily Cal. Stds.....Prior to samples
Samples *

*Any given 12-hour period may contain various quality controls. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.

Prep QC	Frequency
MB	1 per batch [3]
LCS [1]	1 per batch
MS/MSD's [2]	1 set in batch
Surrogates	Every sample / MB / LCS / MS / MSD

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract....pre-determined by the extraction department.
[2] The sample selection for MS/MSD are rotated among client samples so that various matrix problems may be noted and/or addressed. ....pre-determined by the extraction department.
[3] A batch is not to exceed 20 samples.....pre-determined by the extraction department.

Every attempt is made to run a full extraction batch or at the minimum its associated blank, within the same 12-hour period or next 12-hour period, on the same instrument, to keep the preparation QC as reflective of the entire analysis as possible. If, for TAT reasons, a portion of the batch must be analyzed quickly and far in advance of the rest of the batch, the MB may be re-analyzed with the remainder of the batch. The MS/MSD is analyzed once, with their respective client.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests (Refer to the BNA Extraction SOPs for Methods 3510C, 3550B, 3541 and 3580A for more specific information). Listed below are the holding times and the references that include container and preservation requirements for compliance with SW-846. Holding times prior to extraction are:

Matrix	SW-846 (VTS)[1]	Preservation
Soil/Sediment	14 days	Cool 4 ± 2°C
Water	7 days	Cool 4 ± 2°C
Waste / Oil	14 days	Cool 4 ± 2°C

[1]VTS = verified time of sampling

All samples extracted for BNA analysis are stored in extraction batches, in 1.5 - 2.0 mL screw-capped Teflon-lined vials or appropriately sized ground-glass volumetric, at 4 ± 2°C in the dark until analysis. All extracts are analyzed within 40 days of extraction.

7.3 Sample Preparation

7.3.1 Waters - Samples Extracted by Method 3510C

Samples (waters, aqueous sludges) prepared by Methods 3510C, produce ~1-mL extracts containing both the acid and base/neutral fractions. Dilute the extract up to the final volume of 1-mL in a gas-tight syringe with DCM. The syringe is rinsed a minimum of 5x in clean DCM in between samples to prevent cross contamination.

Samples that are suspected to have high levels of organic contamination mat be screened prior to analysis. Based on the screener result, an initial dilution may be performed.

400 uLs extract + 4 uLs Internal Standard.

This leaves 600-uLs extract remaining for secondary dilutions or a repreparation.

7.3.2 Soils, Sediments, Organic Sludges Extracted by SW 3541 / 3580A

These samples prepared by one of the above reference methods; results in one 1-mL extract (0.5 mL extract volume for 3541). The default soil prep method is 3541. Dilute the extract up to the final volume of 1-mL in a gas-tight syringe with DCM. The syringe is rinsed a minimum of 5x in clean DCM in between samples to prevent cross contamination.

Samples that are suspected to have high levels of organic contamination may be screened prior to analysis. Based on the screener result, an initial dilution may be performed.

500 uLs extract + 5 uL Internal Standard for 3580A.
300 uLs extract + 3 uL Internal Standard for 3541.

This leaves ½ volume of the extract remaining for secondary dilutions or a repreparation. Normal waste samples have a final volume of 10 mLs and are prepared as defined above.

7.4 Calibration Standardization

Refer to Section 6.0. Instrument calibration is based on a 12-hour tune or the calibration is completed on an as-needed basis (Section 6.1).

7.5 Preventive Maintenance

Instrumental maintenance can be broken down into two categories: daily and "as required".

7.5.1 Daily Maintenance

- Change injection port septum.
- Check and replace liner.
- Remove column nut, check graphite ferrule - replace if necessary, and trim column.

- Check detector nut and be sure it is tightened completely.
- Replace rinse DCM
- Bake out the GC at 300°C for ~30 minutes

7.5.2 "As Required" Maintenance

Most maintenance is performed on an "as needed" basis, is operator determined and can be categorized as GC or MSD related.

Each required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. Refer to the GC/MSD Maintenance Manual for procedures, references, and documentation of maintenance actions. All maintenance or significant occurrences are entered into the instrument maintenance logbook with a reference number.

7.5.2.1 GC related

- Clean injection port, swab out inlet with Methanol.
- Sonicate/clean or replace gold seal, washer, and reducing nut.
- Change column. Use DB5MS, 30m-x 0.25 mm ID x 0.50um, bonded-phase silicone coated fused-silica capillary column. Condition the column as follows:

Set oven program to:	initial temp = 40°	initial time = 30 min.
Rate= 1°/min	final temp = 300°	final time = 60 min.

NOTE: Be sure there is flow through the column.

- Check Helium flow rate to inlet and the stability of the Electronic Pressure Control (EPC); clean or replace injection port lines.
- Change gas cylinder once 500 psi is attained, change oxy-trap every other cylinder, check for leaks.
- Clean liners and store in sylon.

7.5.2.2 MS Related

- Check vacuum, and interface nut;
- Clean source and anything associated with that activity. Refer to the MSD maintenance manual for details concerning source disassembly, cleaning, and reassembly.

7.6 Sample Analysis

7.6.1 Analyze 1 uL of sample by the method listed in Attachment 1. The method outlined in Attachment 1 is an example and minor adjustments often have to be made to the method in regards to scan start/ending time. The analysis method should remain constant corresponding to the conditions of the calibration verification standard(s).

7.6.2 After quantitation, any extract showing analytes of interest exceeding the calibration range is appropriately diluted and re-analyzed.

7.6.3 Extracts are stored after analysis at 4±2°C until disposal. Extracts are kept for a minimum period of 1 year from the date of analysis.

7.7 Documentation

7.7.1 Data Management

Although there is a substantial amount of space available to both BNA's and VOA's during busy periods, the system can fill rather quickly. As an estimate, a maximum of about 3 months (per instrument) can be kept on the system at one time. No data is removed from the system until it has been archived on at least one full back up and a number of differential back-ups.

7.7.2 Archival of Data

Back-ups are scheduled to occur automatically through the ARCServe software. Once a week, a full back-up of all data generated during the week is completed. These run at 1:00 AM to minimize impact. All of these tapes are permanent saves. A full system back-up, minus data, is also completed once a week and the tape preserved. The system maintains a database, or logs, of each back-up session. Successful completion of each back up can be verified by accessing the job report logs in ARCServe. Any missed jobs can be re-scheduled and completed in the morning of the following day. Currently, archival and retrieval of all GC/MS data is handled by the IS Department.

8.0 QUALITY CONTROL

8.1 QC Summary

The department will review the quality controls as follows:

8.1.1 At least one MB will be included in each extraction set (maximum of 20 samples). The MBs will be examined to determine if contamination is being introduced in the laboratory. The corrective action policy for MBs is found in Section 8.2.10.

8.1.2 One LCS will be included in each extraction set (maximum of 20 samples). Two LCS samples will be extracted when insufficient sample is available for the MS/MSD. When two LCS's are performed, they will be examined to determine both precision and accuracy. The corrective action policy for LCS samples is found in Section 8.2.11.

Accuracy will be measured by the percent recovery (%R) of the LCSs. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy.

8.1.3 Surrogate compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.2 Corrective Action

There are two corrective action reports in use in the GC/MS BNA analysis: the Sample Corrective Action Report and the sequence log.

8.2.1 DFTPP

If in the event that upon analysis of DFTPP at 50 ng/uL the ion ratios do not meet the acceptance criteria listed in Table 3, the analysts must perform the following:

8.2.1.1 All corrective actions taken must be documented in the sequence log and may include some but not all of the following:

- confirm that the DFTPP standard solution has not degraded or has not expired;
- inspect the GC and/or MS system for malfunction or problem that may suggest the need for instrument maintenance as listed in Section 7.5; and
- retune using PFTBA and reanalyze the DFTPP at 50 ng/uL.

8.2.1.2 Upon reanalysis of DFTPP that meets the acceptance criteria listed in Table 3, the demonstration of control must be documented in the sequence log. It is the policy of the BNA lab that: **No further analysis will be performed using this method until an acceptable DFTPP analysis has been successfully performed.**

8.2.2 Initial Calibration

8.2.2.1 If in the event the initial calibration does not pass acceptance criteria as specified in Section 6.4.1.3, the analyst must perform the following:

The corrective actions may include some but not all of the following:

- confirm that the initial calibration standard solutions have not degraded or have not expired; prepare new standards if suspect; reanalyze DFTPP and the initial calibration;
- inspect the GC and/or MS system for malfunction or problem that may suggest the need for instrument maintenance as listed in Section 7.0; perform any necessary maintenance or correct instrument problem; rerun DFTPP and initial calibration; and
- inspect data and rerun any or all of the initial calibration curve points.

If in the event that samples must be analyzed using an out-of-control initial calibration due to analysis hold time considerations or a client specified acceptance of this initial calibration due to a rush TAT, the qualification of data effected must be documented in the sequence log and addressed in the case narrative and/or Non-Conformance Memo (NCM).

8.2.3 Calibration Verification

If in the event the calibration verification does not pass acceptance criteria as specified in Section 6.5.1, the analyst must follow the TNI standard EL-V1M4-2009, Section 1.7.2 , which states:

" If the continuing calibration verification results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial calibration shall be performed".

Immediately running an ICAL without correcting the problem would not be productive. It could result in analyzing samples on a poor ICAL or on an instrument that is not functioning correctly. If two CCV's fail and corrective action was performed then, two acceptable consecutive CCV's must

be obtained to prove the corrective action fixed the problem. If an acceptable CCV can not be obtained and the instrument appears to be stable then a new initial calibration should be analyzed.

8.2.3.1 All corrective actions taken must be documented in the Corrective Action / Qualification Report (CHI-22-21-039) and may include some but not all of the following:

- When a CCV exceeds its recommended acceptance criteria, inspect the system to determine the cause and restore original conditions. Reanalyze the standard or prepare a new standard. Document all actions / reasons in the comment section of each runlog. **Under no circumstances is a CCV to be re-injected prior to completing the aforementioned reviews; or automatically injecting 2 CCV's.**
- There are various types of instrument maintenance that should not automatically require recalibration. Examples include changing/adjusting: septa; compressed gas cylinders; syringes; moisture, hydrocarbon or oxygen traps; split seals; column fittings; inlets; or filaments. Other procedures include breaking off or changing a guard column or cleaning the inlet. Whenever such procedures are performed, the analyst must demonstrate that the results for a CCV meet the prescribed acceptance limits before analysis of any samples. Otherwise, recalibration is required.
- If the % recovery(s) are still out-of-control, a new ICAL sequence is required, and all samples that were injected after the last in control standard must be reanalyzed.
- Maintenance procedures that automatically require recalibration include: changing the column; changing the entrance lens, draw out lines or repeller; cleaning the MS source; changing the electron multiplier, ion source chamber or injector port. Whenever such procedures are performed, the analyst must perform a new ICAL that meets the method requirements. Changing or replacing the column will also require that the retention time windows will need to be re-determined.

8.2.3.2 Upon reanalysis of the calibration verification that meets the acceptance criteria listed in Section 6.5.1, the demonstration of control must be documented in the sequence log.

8.2.3.3 If in the event that samples are analyzed using an out-of-control calibration verification due to analysis hold time considerations or a client specified acceptance of this calibration verification, the qualification of data effected must be documented in the sequence log and addressed in the case narrative and/or Non-Conformance Memo (NCM).

8.2.4 Internal Standard Areas

Internal standard responses (area counts) must be monitored in all samples, spikes and blanks. If the area counts for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the HSL calibration verification analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be addressed in a Non-Conformance Memo (NCM).

8.2.5 The Sample Corrective Action Report

The Sample Corrective Action Report (CAR) (Attachment 5), is attached to the back of every chain-of-custody and is used to provide corrective action for the analysis of samples, MS/MSD's, MB's, and LCS's. The Sample CAR is a tracking system for the decision making process in regards to corrective action for dilutions, internal standards, surrogate recoveries or any other occurrence that is identified in the aforementioned analyses. The corrective action for a set of samples is tracked using the batch on the Sample CAR. The decision for a corrective action on a sample are initialed and dated by the analyst. All sample analyses are tracked on this form even if there is not a corrective action required.

Out-of-control situations are identified upon initial data review/reduction immediately following sample analysis. The analyst must perform the following steps before an out-of-control situation is identified. The analyst should:

- rule out any instrument malfunction or mis-injection of the sample;
- verify that all peaks of interest are integrated completely;
- verify that all calculations are correct;
- verify concentrations of surrogates/spikes in the spiking solutions are correct;
- verify that the sample was prepared correctly and that the correct amount of ISTD was added;
- verify that the ISTD and surrogate areas are reasonable; and
- verify that if a dilution was made was it prepared correctly

In addition, an instrument sequence log is printed, reviewed, signed and bound in a log daily for each instrument. The instrument sequence log is used to provide corrective action for the analysis of DFTPP, Initial Calibrations, Calibration Verifications and Qualification of Data (Attachment 5). The description of the situation, the corrective actions taken, the demonstration of control, and any data qualification are documented.

8.2.6 Dilution Corrective Action Policy

Upon the identification of an out-of-control event concerning a sample needing either an alternate initial dilution or a secondary dilution, the corrective action decision is documented on the Sample CAR. The analysis of the dilution is also recorded on the Sample CAR, indicating that the dilution was successful, thus demonstrating a return to control, or that additional dilutions may be required.

An alternate initial dilution may be required if in the opinion of the analyst that in the first analysis the sample background matrix has saturated the detector. The sample would require a reanalysis at a dilution and is considered the initial analysis. The necessary initial dilution level is documented on the Sample CAR with the initials of the analysts and the date.

A secondary dilution on a sample must be performed if the on-column quantitation of any compound exceeds the linear range of the initial calibration curve (80-ng on-column). The necessary secondary dilution level is documented on the Sample CAR with the initials of the analyst and the date the sample on the report.

8.2.7 Screening of Samples

Samples suspected of being high in target and non-target compounds may be screened on a GC/MS instrument. Initial sample dilutions are determined from this screening analysis. All efforts are made to run all samples undiluted to achieve the lowest reporting limits possible. However, severe matrix effects will result in the laboratory running initial dilutions to obtain the best chromatography possible and reduce damage to our instrumentation and columns. Severe matrix effects can be defined as any unknown peak which is at least two times the peak height of its applicable internal standard or any group of peaks that results in peak heights one time above their respective internal standard. The laboratory will use professional judgment in determining initial dilutions. The laboratory will make all attempts to obtain surrogate recoveries in all initial dilutions but generally they are not achievable in dilutions of 40x or greater.

8.2.8 Internal Standard Areas / Retention Times Corrective Action Policy

Internal standard responses (area counts) must be monitored in all samples, spikes and blanks. If the area counts for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the HSL calibration verification analyzed that day, corrective action must be taken. Some QAPPs require comparison to the areas in the midpoint of the HSL initial calibration. QAPP requirements will supersede the SOP policy. The internal standards in the samples will not vary in retention time by more than ±30 seconds from HSL initial calibration mid-point calibration standard. Upon the identification of an out-of-control event in a sample analysis (in regards to internal standard areas or retention times), the corrective action is documented on the Sample CAR with the decision whether to reanalyze the sample, initialed and dated.

8.2.8.1 The decision to reanalyze a sample is based on the following criteria:

- All samples, MS/MSD's, MB, and LCS samples will be reanalyzed due to a shift in the retention time of the internal standards.
- A secondary dilution is considered a reanalysis for a sample with internal standard areas outside of the acceptance limits. A reanalysis is not required on a secondary dilution with internal standard areas outside of acceptance limits.
- If the sample and the MS/MSD on that sample exhibit similar internal standard area patterns, then the sample does not require a reanalysis.
- Samples with out-of-control internal standard areas may not require a reanalysis if in the opinion of the analyst and with the approval of the supervisor or section manager the following situation(s) occur:
- there is obvious matrix interference in the samples causing the internal standard areas to be out-of-control.
- there are no compounds detected in the sample greater than the reporting limit that uses the out-of-control internal standard for quantitation.
- there is approval given by supervisor, the section manager, the Project Manager or the client that a reanalysis is unnecessary.

8.2.8.2 The results of a sample reanalysis due to out-of-control internal standards are documented on the Sample CAR.

If upon reanalysis of the sample, the internal standards meet the acceptance criteria, the documentation of return to control is recorded on the Sample CAR, and the reanalysis is reported as the initial analysis.
If upon reanalysis of the samples the internal standard is still out-of-control, this is documented in the Sample CAR. Both the initial and reanalysis is reported on the sample. All corrective action decisions are addressed in the case narrative and/or Non-Conformance Memo (NCM).

8.2.9 Surrogate Recovery Corrective Action Policy

The surrogate recovery QC limits for both water and soil matrices are found in Table 2. Upon identification of an out-of-control event in regards to surrogate recoveries, the corrective action will be documented in the Sample CAR and the decision of whether to re-extract the sample is initialed and dated by the analyst. The sample re-analysis or re-extraction/reanalysis is also documented on the Sample CAR. This provides a documentation of a return to control or situation that requires addressing in the case narrative and/or Non-Conformance Memo (NCM).

8.2.9.1 The decisions regarding corrective action for out-of-control surrogate recoveries are made using the following policies:

- If any surrogates are outside of limits in the MB, the MB may be reanalyzed. If the surrogate recoveries are also out-of-control in the reanalysis, the entire extraction batch will have to be re-extracted and reanalyzed.
- LCS samples do not require re-extraction due to out-of-control surrogate recoveries nor do associated samples require re-extraction.
- MS/MSD samples do not require re-extraction due to out-of-control surrogate recoveries.
- If one surrogate in any one sample is out of limits and all surrogate recoveries are greater than 10%, then the sample does not require re-extraction. If one acid and one base-neutral is out of limits in any one sample analysis and all surrogate recoveries are greater than 10%, then the sample does not require re-extraction.
- If the sample with the out-of-control surrogates is the same sample on which the MS/MSD was performed, and the MS/MSD show the same recovery pattern, then a re-extraction on the sample is not required.

A re-analysis of the sample is suggested before continuing. If an entire batch is showing the same pattern, it may be related either to matrix or a common preparation problem or error. If re-analysis gives the same result, a common error or matrix effect is suspected and the sample(s) is/are re-extracted and re-analyzed.

8.2.9.2 The following policy applies to re-extracted/reanalyzed samples:

- If the surrogates are within the QC limits, then an in-control situation has been demonstrated and only the second analysis is reported. The laboratory makes every attempt to re-extract samples within extraction holding times. If the re-extraction occurs beyond the hold time, then both the initial and the re-extracted results are reported on the samples.
- If the surrogates are still out-of-control, both the initial and re-extracted results are reported on the samples. An NCM must be completed and approved prior to the reporting of sample data.

8.2.10 Contamination in the Method Blank

If the MB is/appears to be contaminated with compounds then the corrective action is documented in the Sample CAR. The decisions regarding the corrective action on a contaminated method blank are made using the following course of action:

- Reanalyze the MB on another instrument (one that has demonstrated a clean blank or sample).
- If contamination appears again, re-prepare the MB from the remaining extract. Analyze the re-prepared MB on the first instrument again.
- If the MB was found to be uncontaminated on the second instrument or after re-preparing (from the remaining extract) the source of contamination is a) in the first instrument or b) was introduced during the sample preparation just prior to analysis. Report only these results for the method blank.
- If upon repreparation/reanalysis, the MB is contaminated, the contamination was probably introduced during the extraction procedure. The level of corrective action is based on the concentration of the compound contaminant(s) found in the MB. If the on-column amount for any compound is greater than the reporting limit or greater than 5x the reporting limit for the phthalate ester, than the samples associated with this MB must be re-extracted.

- A situation may occur where the samples associated with a contaminated MB may not require re-extraction when the contaminating compounds are not detected on the samples above the reporting limit which is approved by the supervisor or the section manager, the QA department, the Project Manager or the client.
- The re-extracted samples are reported as the initial analysis and the first extraction set need not be analyzed. The laboratory makes every attempt to re-extract samples within extraction holding times. If the re-extraction occurs beyond the hold time, then both the initial and the re-extracted results are reported on the samples.
- Any hold time violation requires that a NCM be filled out and submitted to the project manager for approval before any results are reported.

8.2.11 LCS Recoveries

In-house QC limits from the analysis of the TCL BNA LCS spike (see Section 5.3) are generated annually. Upon identification of an out-of-control event in regards to LCS recoveries, the corrective action will be documented in the Sample CAR and the decision of whether to re-extract the associated samples is initialed and dated by the analyst. The sample reanalysis or re-extraction/reanalysis is also documented on the Sample CAR. This provides a documentation of a return to control or situation that requires addressing in the case narrative and/or Non-Conformance Memo (NCM).

8.2.11.1 The LCS limits are based on the mean recovery +/- 3 standard deviations, therefore, it is statistically quite likely that there will be exceedances of the limits for a few analytes when a large number of compounds are included in the LCS. Therefore a number of individual analytes are allowed to exceed the LCS control limit before the LCS as a whole is considered to have failed. The control limit for a marginal exceedance is mean +/- 4 standard deviations which results in a larger range of +/-10% outside the in-house generated limits. The number of marginal exceedances allowed is based on DOD guidance: Compounds that are identified as contaminants of concern for a project are not included in the marginal exceedance allowance.

Number of analytes in LCS	Number of marginal exceedances allowed
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

Note: For samples received from South Carolina, the full list of routinely spiked analytes will be reported to the client. The controlling compound spike list will be agreed upon with the client at the time of project set up. The use of marginal exceedances is not allowed for the state of South Carolina.

If the LCS recovery is outside of the in-house QC limits, the following actions may be taken concerning reporting or re-extracting the associated samples:

- If there is a MS/MSD extracted on a sample in the same extraction set and the spike recoveries are acceptable within these samples, then the samples do not require re-extraction and the corrective action is addressed in the case narrative and/or Non-Conformance Memo (NCM)

- If there was not a MS/MSD extracted in the same extraction set and the LCS/LCD recoveries are above the QC limits, then the associated samples require re-extraction only if the spike compounds are detected in the samples above the reporting limits.
- If there was a MS/MSD extracted in the same extraction set; the spike recoveries are outside of QC limits within these samples; and the LCS recovery was above the QC limits, then the associated samples require re-extraction only if the spike compounds are detected in the samples above the reporting limits.
- If the spike recoveries in the MS/MSD are outside of the QC limits and the LCS recovery was below the QC limits, then the samples associated with the LCS sample are re-extracted.
- Samples may require re-extraction if contaminants of concern identified by the client or QAPP fail in the LCS, regardless of the number of marginal exceedence's.
- Other situations where the LCS recovery is outside the QC limits will be evaluated on a case by case basis and the decision to re-extracted or report the associated sample will be made by the supervisor or section manager with the approval of the QA department, the Project Manager or the client.
- If the recovery in the LCS sample is outside of the QC limits, the LCS may be re-prepared using the remaining extract and reanalyzed on the same instrument or another instrument. If all recoveries are acceptable in the reanalysis, then the second analysis is reported as the initial analysis, the method is considered to be within control and the associated sample does not require re-extraction.

8.2.11.2 The following policies apply to samples re-extracted/reanalyzed due to out-of-control LCS spike recoveries:

- If the re-extraction occurs within the extraction holding times, then only the second analysis on the samples is reported. The laboratory makes every attempt to re-extract samples within extraction holding times.
- If the re-extraction occurs beyond the hold time, then both the initial and the re-extracted results are reported on the samples. An NCM must be completed and approved prior to the reporting of sample data.

All corrective action is documented on the Sample CAR with the decision whether to reanalyze the LCS or to re-extract all associated samples; initialed and dated by the analyst. The sample reanalysis is also documented on the Sample CAR.

8.2.12 Matrix Spike (MS) / MS Duplicate (MSD)

In MS/MSD analyses, the spike recoveries and RPD values (Table 1) function to illustrate and note possible matrix effects on the efficiency and completeness of analyte extraction. Out-of-control events on MS/MSD's are documented on the Sample CAR and initialed and dated by the analyst. Sample re-extraction is not performed due to out-of-control MS/MSD recoveries or RPD values; therefore, no return to control can be documented. The Sample CAR serves as a mechanism to address the out-of-control events in the case narrative and/or Non-Conformance Memo (NCM). Corrective action in these situations is not used to suggest re-extraction. The corrective action decisions for out-of-control events on MS/MSD samples are driven by the acceptance/rejection of the LCS spike recovery. (Section 8.2.11).

8.2.13 Any other occurrences that require addressing in the case narrative and/or Non-Conformance Memo (NCM) are documented either in the Sample CAR and the corrective action is evaluated on a case by case basis.

8.2.14 Each instrument has a Maintenance Logbook. Major instrument maintenance and problem resolutions are documented in the maintenance logbook. The logbook provides for a description of the maintenance or repair performed and a corrective action for a return to control event, which is initialed and dated by the analyst.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Chrom software produces a Total Ion Chromatogram (TIC); quant report and background subtracted spectra of analytes. For those clients requiring it, tentatively identified compound (TIC) searches are also performed. The data system will produce library search report, which includes unknown quantitation report and unknown mass spectra with the TICs with the highest probability. All data for each sample is kept together.

9.2 Quantitation of Compounds

Quantitation of the compounds and response factors are performed by the data system, but can be accomplished as follows:

9.2.1 Water

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_o)(V_i)}$$

Where:
A_x = area of characteristic ion for compound of interest
I_s = amount of internal standard injected (ng) (ISTD).
V_t = volume of total extract (uLs). This includes a measured final volume
A_{is} = area of characteristic ion of designated internal standard
RF = Average Response Factor from Initial Calibration
V_o = volume of water extracted (mLs)
V_i = volume of extract injected (uLs)

9.2.2 Soil, Sediment, Waste

$$\text{Concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

All variables are the same as in the water calculation in Section 9.2.1 with the addition of the following:

W_s = weight of sample extracted or diluted (g)
D = (100 - % moisture in sample) / 100 (or 1 for wet weight basis)

A 2x dilution factor must be taken into account whenever the soil samples have undergone a GPC clean up.

NOTE: All dry weight corrections are made in TALS AD batch at the time the data is processed.

9.2.3 Accuracy

$$\%R = \frac{(A_T - A_0)}{A_F} \times 100$$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.2.4 Precision

$$\% D = \frac{|B_1 - B_2|}{B_1} \times 100$$

$$RPD = \frac{|B_1 - B_2|}{(B_1 + B_2)/2} \times 100$$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCD – when performed)

9.2.5 Quantitation by Method 8270D

The samples analyzed by Method 8270D will be quantitated using the initial calibration. For compounds that are calibrated using a linear regression curve, the following equation is used to determine the on-column amount of a compound:

Methods 8000/8270D require the use of linear or higher order calibration curves for those compounds exceeding 20%.

The following equations apply:

Linear Regression: $y = a_0 + a_1 * x$
Weighted Linear Regression: $y = a_1 * x + a_0$
Quadratic Curve: $y = a_o + (a1 * x) + (a2 * x^2)$

Where: $x = Area_{UNK}/Area_{ISTD}$
$y = Amount_{UNK}/Amount_{ISTD}$
a_1 = slope
a_0 = y-intercept

The equation for weighted linear regression follows the linear regression but introduces a weighting factor for the slope and y-intercept. The User manually enters the weighting factor into the method as: 1/Amt or $1/Amt^2$

The quantitation of compounds using either a linear regression is performed by the Chrom software and has been confirmed to be accurate to obtain the on-column $Amount_{UNK}$ of a compound reported on the quantitation report. To obtain the final concentration the following equations are used:

Waters Concentration (µg/L) = $(Amount_{UNK})(V_t)/(V_o)(V_i)$

Soils Concentration (µg/L) = $(Amount_{UNK})(V_t)/(V_i)(W_s)(D)$

* The variables V_t, V_o, V_i, W_s, and D are defined in Section 9.2.1 and 9.2.2.

9.3 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the Chrom system. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the unknown quantitation report produced by the data system. The nearest ISTD should be used. The RF is assumed 1. The concentration is therefore an estimate.

$$\text{Total ng of TIC} = \frac{\text{area of TIC x ng of its ISTD}}{\text{area of that ISTD}}$$

9.4 Operator Data Reduction/Review

The operator reviews the data and 1) makes judgments concerning the "realness" of those compounds found and 2) makes judgments concerning the identification of the TICs and 3) modifies the output to confirm with those judgments to produce a data package reflective of those decisions. All initial data reduction/review is performed by the analyst in Chrom review before any reports are generated.

9.5 Initial Review

Operator determines that the analysis itself is acceptable. This means:

9.5.1 QC samples meet acceptable limits. See Section 8.2 for corrective action regarding sample analysis.

NOTE: Table 1 contains the laboratory's internally-derived statistical control limits for the MS/MSDs, the LCS, and the surrogates. The limits used to assess precision and accuracy as well as the number of compounds being used for bench level control may vary with client, QAP, project, etc... This information is transmitted to the bench via the COC: kick-off meetings, tech profiles, etc...

- Glassware blank (MB) is clean, ALL surrogates are within control limits. A clean MB means no compounds above the reporting limit; except for the phthalates that must be less than 5x the reporting limit.
- All surrogates in the samples are within control limits or at most one surrogate in each fraction is out. No surrogate can be <10%. All surrogates must be within control limits for all MBs.
- All MS/MSD and LCS recoveries are within in-house generated control limits specified in Table 1 for the spike compounds in the HSL BNA LCS spike identified in Section 5.3.

9.5.2 The sample does not require any further dilutions or analysis at a more concentrated level. Visually confirm complete integration for any large and/or saturated compound found. Dilutions are made to keep the compounds in the upper half of the calibration range. MS/MSD or LCS samples are never diluted to get spiked or non-spiked analytes within the calibration range.

9.5.2.1 The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc.)

9.5.2.2 Identification of compounds. The following criteria are used in the positive identification of compounds. **These are guidelines, they are not absolute.**

- "elution of component at the same relative retention time as the standard component."

The elution times should compare within 0.06 min. The standard MUST be run on the same 12-hour period as the sample. If co-eluting analytes interfere with comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

- "correspondence of the sample component and standard component mass spectrum."

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) MUST be present in the sample.

The relative intensities of the above ions must agree within ±30%, between the standard and sample. If an ion has 50% intensity in the standard, the corresponding ion must be between 20% and 80% in the sample.

Ions >10% in the sample but not present in the standard must be considered and accounted for. If a compound cannot be verified by the above, **but in the operator's technical judgment, the ID is correct**, it is reported as such.

9.5.2.3 Once all positive identification is made, the file is modified to reflect these decisions. All analytes at concentrations less than the reporting limit are automatically flagged with a 'J'.

9.5.2.4 Identification of TIC's

In general, up to as many as 20 non- compounds are tentatively identified by the data system and operator. The data system provides the operator with a subtracted mass spectrum of the unknown and the mass spectra of the first 3 matches. Molecular formulas, molecular weights, and CAS #'s are included. The following **guidelines** are used:

9.5.2.4.1 Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within ±20%.
- Molecular ions in the reference should be in the samples.
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard.
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds.
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds.
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula, or weight.

9.6 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must follow these guidelines:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data along with the reason for integration and analyst who performed the integration.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.
- Manual integrations shall follow CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations located in Attachment 8. An example of one such integration and documentation is included in the attachment.

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of compound detected.

- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

The analysts review all integrations. Any integration that takes place for compounds are routinely included in the L4 data packages. If integrations for level 2 reports are required to satisfy client deliverables, they can be provided, but will not be routinely added. Manual integrations may be documented in the case narrative and/or Non-Conformance Memo (NCM) if so required, however, references to this SOP will be used for explanations, and any further documentation beyond initials and dates will not be performed.

9.7 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analysts transfer the data into the TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 6) and sets the batch status in TALs to 2nd level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then approved or "sunshined". Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*
Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

Note: The entire sample tracking system can be summarized in Attachment 7. The complete analysis scheme can be summarized in Sections 7.1.1, 7.1.2 and in Attachment 7.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out:

- Waste from this procedure will enter the Methylene Chloride (DCM) waste stream.
- Expired and single component standards will be turned over to the EHSC or Waste Technician.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Sections 1.0.

13.0 ATTACHMENTS

Table 1: Characteristic Ions for Semi-Volatile Organic Compounds
Table 2: Example: Reporting Limits (RLs); Internal Statistical Control Limits for MS/MSDs, LCSs, Surrogates for Water and Soil/Sediment Samples
Table 3: DFTPP Key Ions and Ion Abundance Criteria
Table 4: Calibration Check Compounds and System Performance Check Cmpds.
Table 5: Internal Standards with Corresponding Analysts Assigned for Quantitation.

Attachment 1: Example: BNA Method for Standards and Samples
Attachment 2: CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points
Attachment 3: Example: Initial Calibration - Form 6
Attachment 4: Example: Calibration Verification - Form 7
Attachment 5: Example: Sample Corrective Action Report / Run Log Report / Instrument Maintenance Log
Attachment 6: Example: Data review checklist
Attachment 7: Example: Analysis Scheme and Sample Tracking Flowcharts
Attachment 8: CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations and example documentation.
Attachment 9: Example: ICV recoveries

Appendix A: DOD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1; and Table F-4

14.0 REVISION HISTORY

- Revision 04 updated on 11/28/12
- Annual Review
- Section 8.2.3 and 8.2.3.1 added reference to specific language in the TNI Standard EL-V1M4-2009 section 1.7.2.

Table 1. Characteristic Ions for Semi-Volatile Organic Compounds

Compound	Approx. Retention Time (Min)	Primary Ion	Secondary Ion(s)
Acenaphthene	8.58	154	153, 152
Acenaphthene-d_{10} (I.S.)	8.54	164	162, 160
Acenaphthylene	8.37	152	151, 153
Acetophenone	5.66	105	77, 51
2-Acetylaminofluorene	12.22[a]	181	180, 182, 223
Aniline	4.94	93	66, 65
Anthracene	10.19	178	176, 179
4-Aminobiphenyl	9.99[a]	169	168, 170
Aramite	11.5[a]	63	185, 191, 187
Benzidine	11.30	184	92, 185
Benzoic Acid	6.32	122	105, 77
Benzo(a)anthracene	12.39	228	229, 226
Benzo(b)fluoranthene	13.38	252	253, 125
Benzo(k)fluoranthene	13.40	252	253, 125
Benzo(g,h,i)perylene	15.71	276	138, 277
Benzo(a)pyrene	13.74	252	253, 125
Benzyl alcohol	5.37	108	79, 77
Bis(2-chloroethoxy)methane	6.31	93	95, 123
Bis(2-chloroethyl)ether	4.99	93	63, 95
2,2'-oxybis(1-Chloropropane)	5.53	45	77, 121
Bis(2-ethylhexyl)phthalate	12.37	149	167, 279
4-Bromophenyl phenyl ether	9.66	248	250, 141
Butylbenzylphthalate	11.92	149	91, 206
sec-Butyl-4,6-dinitrophenol	10.24	211	212, 240, 163
4-Chloroaniline	6.67	127	129
Chlorobenzilate	11.88	139	251, 253, 141
2-Chloronaphthalene	7.90	162	127, 164
4-Chloro-3-methylphenol	7.71	107	144, 142
2-Chlorophenol	5.05	128	64, 130
4-Chlorophenyl phenyl ether	9.13	204	206, 141
Carbazole	10.34	167	166, 139
Chrysene	12.42	228	226, 229
Chrysene-d_{12} (I.S.)	12.40	240	120, 236
Diallate	9.65	43	86, 234
Dibenz(a,h)anthracene	15.27	278	139, 279
Dibenzofuran	8.76	168	139
Di-n-butylphthalate	10.67	149	150, 104
1,3-Dichlorobenzene	5.20	146	148, 111
1,4-Dichlorobenzene	5.25	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	5.23	152	150, 115
1,2-Dichlorobenzene	5.44	146	148, 111
3,3'-Dichlorobenzidine	12.35	252	254, 126
2,4-Dichlorophenol	6.42	162	164, 98
2,6-Dichlorophenol	6.67_a	162	164, 98
3,3'-Dimethylbenzidine	12.00	212	211, 213, 196
meta-Dinitrobenzene	8.34	168	76, 75, 92
1,4-Dioxane	2.68	88	58, 87
Diethylphthalate	9.06	149	177, 150
p-Dimethylaminoazobenzene	11.56_a	120	225, 77
7,12-Dimethylbenz(a)anthracene	13.50_a	256	241, 257
a,a-Dimethylphenethylamine	6.60_a	58	91, 42
2,4-Dimethylphenol	6.19	122	107, 121

Table 1. Characteristic Ions for Semi-Volatile Organic Compounds

Compound	Approx. Retention Time (Min)	Primary Ion	Secondary Ion(s)
Dimethylphthalate	8.27	163	194, 164
4,6-Dinitro-2-methylphenol	9.25	198	51, 105
2,4-Dinitrophenol	8.62	184	63, 154
2,4-Dinitrotoluene	8.79	165	63, 89
2,6-Dinitrotoluene	8.35	165	63, 89
Diphenylamine	9.24[a]	169	168, 167
1,2-Diphenylhydrazine	9.31	77	105, 182
Di-n-octylphthalate	12.88	149	167, 43
Ethyl Methanesulfonate	3.66[a]	79	109, 97
Fluoranthene	11.22	202	101, 203
Fluorene	9.12	166	165, 167
2-Fluorobiphenyl (surr.)	7.78	172	171
2-Fluorophenol (surr.)	4.04	112	64
Hexachlorobenzene	9.82	284	142, 249
Hexachlorobutadiene	6.78	225	223, 227
Hexachlorocyclopentadiene	7.60	237	235, 272
Hexachloroethane	5.76	117	201, 199
Hexachlorophene	13.60	196	198, 209, 406
Hexachloropropene	6.73	213	215, 211, 143
Indeno(1,2,3-cd)pyrene	15.28	276	138, 227
Isophorone	6.09	82	95, 138
Isosafrole	7.75	162	161, 131, 78
Methapyrilene	11.22[a]	58	97, 72, 191
3-Methylcholanthrene	15.12[a]	268	253, 267
Methyl Methanesulfonate	3.55[a]	80	79, 65
2-methylnaphthalene	7.35	142	141
2-Methylphenol (o-cresol)	5.49	108	107, 79
4-Methylphenol (m&p-cresol)	5.63	108	107, 79
Naphthalene	6.60	128	129, 127
Naphthalene-d_8 (I.S.)	6.58	136	68
1,4-Naphthoquinone	8.33[a]	158	130, 104, 102
1-Naphthylamine	8.77[a]	143	115, 116
2-Naphthylamine	8.99[a]	143	115, 116
2-Nitroaniline	8.03	65	92, 138
3-Nitroaniline	8.50	138	108, 92
4-Nitroaniline	9.20	138	108, 92
Nitrobenzene	5.85	77	123, 65
Nitrobenzene-d_5 (surr.)	5.82	82	128, 54
2-Nitrophenol	6.18	139	109, 65
4-Nitrophenol	8.66	139	109, 65
5-Nitro-o-toluidine	9.24	77	152, 106, 79
4-Nitroquinoline-1-oxide	11.25	190	160, 128, 89
N-Nitroso-di-n-butylamine	7.15[a]	84	57, 41
N-Nitrosodimethylamine	2.97	42	74, 44
N-Nitrosodiethylamine	4.27	42	102, 56, 57
N-Nitrosodiphenylamine	9.28	169	168, 167
N-Nitrosodi-n-propylamine	5.68	70	42, 101, 130
N-Nitrosomethylethylamine	3.67	42	88, 43, 56
N-Nitrosomorpholine	5.88	56	88, 43, 56
N-Nitrosopiperidine	5.94	42	116, 86
N-Nitrosopyrrolidine	5.66	100	41, 68
Pentachlorobenzene	8.79[a]	250	252, 248
Pentachloronitrobenzene	9.78[a]	295	237, 142

Table 1. Characteristic Ions for Semi-Volatile Organic Compounds

Compound	Approx. Retention Time (Min)	Primary Ion	Secondary Ion(s)
Pentachlorophenol	9.99	266	264, 268
Perylene-d_{12} (I.S.)	13.79	264	260, 265
Phenacetin	9.63[a]	108	109, 179
Phenanthrene	10.15	178	179, 176
Phenanthrene-d_{12} (I.S.)	10.12	188	94, 80
Phenol	4.89	94	65, 66
Phenol-d_5 (surr.)	4.88	99	42, 71
p-Phenylenediamine	7.44	108	80, 52, 54
2-Picoline	3.25[a]	93	66, 92
Pronamide	9.87[a]	173	175, 145
Pyrene	11.41	202	200, 203
Pyridine	2.97	79	52, 51
Safrole	7.24	162	161, 131, 104
Terphenyl-d_{14} (surr.)	11.52	244	122, 212
1,2,4,5-Tetrachlorobenzene	7.58[a]	216	214, 218
2,3,4,6-Tetrachlorophenol	8.85[a]	232	230, 131
2,4,6-Tribromophenol (surr.)	9.43	330	332, 141
1,2,4-Trichlorobenzene	6.53	180	182, 145
2,4,5-Trichlorophenol	7.73	196	198, 200
2,4,6-Trichlorophenol	7.69	196	198, 200
o-Toluidine	5.90	106	107, 77, 56
Sym-Trinitrobenzene	9.87	75	213, 74

Table 2.

Example: Reporting Limits (RLs); Internal Statistical Control Limits for MS/MSDs, LCSs, Surrogates for Water and Soil/Sediment Samples (043-001 to 043-012)

- RLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, RLs will be higher based on the percentage moisture in each sample.
- Sample RLs are highly matrix-dependent. The RLs listed herein are provided for guidance and may not always be achievable.
- Statistical control limits and MDLs are determined annually and are subject to change. Contact the laboratory for the latest values.

MDL W GCMS BNA 8270D_ICAL 1	Semivolatile Organic Compounds (GC/MS)	8270D

Analyte Description	CAS Number	RL Limit	LOD Limit	MDL Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,2,4,5-Tetrachlorobenzene	95-94-3	5	2.5	1.22	ug/L	42	110	20	42	110	20		
1,2,4-Trichlorobenzene	120-82-1	2	1	0.3	ug/L	34	110	20	34	110	20		
1,2-Dichlorobenzene	95-50-1	2	1	0.29	ug/L	34	100	20	34	100	20		
1,2-Diphenylhydrazine	122-66-7	5	2.5	0.7	ug/L	58	110	20	58	110	20		
1,3-Dichlorobenzene	541-73-1	2	1	0.25	ug/L	34	100	20	32	100	20		
1,4-Dichlorobenzene	106-46-7	2	1	0.27	ug/L	33	100	20	33	100	20		
2,2'-oxybis[1-chloropropane]	108-60-1	2	1	0.3	ug/L	32	110	20	32	110	20		
2,4,5-Trichlorophenol	95-95-4	10	5	2.29	ug/L	63	110	20	63	110	20		
2,4,6-Trichlorophenol	88-06-2	5	2.5	1.1	ug/L	63	110	20	63	110	20		
2,4-Dichlorophenol	120-83-2	10	5	2.28	ug/L	58	110	20	58	110	20		
2,4-Dimethylphenol	105-67-9	10	5	3.34	ug/L	48	110	20	48	110	20		
2,4-Dinitrophenol	51-28-5	20	10	7.43	ug/L	25	148	20	25	148	20		
2,4-Dinitrotoluene	121-14-2	1	0.5	0.3	ug/L	62	119	20	62	119	20		
2,6-Dichlorophenol	87-65-0	5	2.5	0.85	ug/L	57	110	20	57	110	20		
2,6-Dinitrotoluene	606-20-2	0.5	0.25	0.12	ug/L	63	110	20	63	110	20		
2-Chloronaphthalene	91-58-7	2	1	0.34	ug/L	48	110	20	48	110	20		
2-Chlorophenol	95-57-8	5	2.5	0.8	ug/L	50	110	20	50	110	20		
2-Methylnaphthalene	91-57-6	0.5	0.25	0.13	ug/L	39	110	20	39	110	20		
2-Methylphenol	95-48-7	2	1	0.31	ug/L	42	100	20	42	100	20		
2-Nitroaniline	88-74-4	5	2.5	1.08	ug/L	62	121	20	62	121	20		
2-Nitrophenol	88-75-5	10	5	2.14	ug/L	56	110	20	56	110	20		
3 & 4 Methylphenol	15831-10-4	2	1	0.44	ug/L	38	110	20	38	110	20		
3,3'-Dichlorobenzidine	91-94-1	5	2.5	0.94	ug/L	56	111	20	56	111	20		
3-Nitroaniline	99-09-2	10	5	2.29	ug/L	55	113	20	55	113	20		
4,6-Dinitro-2-methylphenol	534-52-1	20	10	4.92	ug/L	58	121	20	58	121	20		
4-Bromophenyl phenyl ether	101-55-3	5	2.5	0.91	ug/L	59	110	20	59	110	20		
4-Chloro-3-methylphenol	59-50-7	10	5	2.2	ug/L	52	111	20	52	111	20		
4-Chloroaniline	106-47-8	10	5	2.1	ug/L	47	110	20	47	110	20		
4-Chlorophenyl phenyl ether	7005-72-3	5	2.5	0.81	ug/L	59	110	20	59	110	20		
4-Nitroaniline	100-01-6	10	5	3.93	ug/L	52	125	20	52	125	20		
4-Nitrophenol	100-02-7	20	10	2.34	ug/L	20	100	20	20	100	20		
Acenaphthene	83-32-9	1	0.5	0.36	ug/L	53	110	20	53	110	20		
Acenaphthylene	208-96-8	1	0.5	0.32	ug/L	56	110	20	56	110	20		
Acetophenone	98-86-2	5	2.5	0.81	ug/L	48	110	20	48	110	20		

Aniline	62-53-3	20	10	3.45	ug/L	29	100	20	29	100	20		
Anthracene	120-12-7	1	0.5	0.32	ug/L	58	110	20	58	110	20		
Benzidine	92-87-5	40	20	20	ug/L	10	100	20	10	100	20		
Benzo[a]anthracene	56-55-3	0.2	0.1	0.044	ug/L	62	110	20	62	110	20		
Benzo[a]pyrene	50-32-8	0.2	0.1	0.056	ug/L	63	110	20	63	110	20		
Benzo[b]fluoranthene	205-99-2	0.2	0.1	0.058	ug/L	60	111	20	60	111	20		
Benzo[g,h,i]perylene	191-24-2	1	0.5	0.42	ug/L	56	123	20	56	123	20		
Benzo[k]fluoranthene	207-08-9	0.2	0.1	0.074	ug/L	59	116	20	59	116	20		
Benzoic acid	65-85-0	20	10	4.56	ug/L	10	100	20	10	100	20		
Benzyl alcohol	100-51-6	20	10	3.05	ug/L	42	110	20	42	110	20		
Bis(2-chloroethoxy)methane	111-91-1	2	1	0.3	ug/L	56	110	20	56	110	20		
Bis(2-chloroethyl)ether	111-44-4	2	1	0.35	ug/L	49	110	20	49	110	20		
Bis(2-ethylhexyl) phthalate	117-81-7	10	5	2.43	ug/L	62	122	20	62	122	20		
Butyl benzyl phthalate	85-68-7	2	1	0.27	ug/L	64	126	20	64	126	20		
Carbazole	86-74-8	5	2.5	0.99	ug/L	62	112	20	62	112	20		
Chrysene	218-01-9	0.5	0.25	0.14	ug/L	62	111	20	62	111	20		
Dibenz(a,h)anthracene	53-70-3	0.3	0.15	0.064	ug/L	59	120	20	59	120	20		
Dibenzofuran	132-64-9	2	1	0.35	ug/L	56	110	20	56	110	20		
Diethyl phthalate	84-66-2	2	1	0.44	ug/L	63	110	20	63	110	20		
Dimethyl phthalate	131-11-3	2	1	0.38	ug/L	65	110	20	65	110	20		
Di-n-butyl phthalate	84-74-2	5	2.5	0.8	ug/L	64	116	20	64	116	20		
Di-n-octyl phthalate	117-84-0	10	5	2.47	ug/L	59	131	20	59	131	20		
Fluoranthene	206-44-0	1	0.5	0.32	ug/L	62	112	20	62	112	20		
Fluorene	86-73-7	1	0.5	0.38	ug/L	53	110	20	53	110	20		
Hexachlorobenzene	118-74-1	0.5	0.25	0.14	ug/L	60	110	20	60	110	20		
Hexachlorobutadiene	87-68-3	5	2.5	1.11	ug/L	28	110	20	28	110	20		
Hexachlorocyclopentadiene	77-47-4	20	10	3.44	ug/L	15	110	20	15	110	20		
Hexachloroethane	67-72-1	5	2.5	0.97	ug/L	29	100	20	29	100	20		
Hexachloropropene	1888-71-7	20	10	3.01	ug/L	23	110	20	23	110	20		
Indeno[1,2,3-cd]pyrene	193-39-5	0.2	0.1	0.084	ug/L	61	117	20	61	117	20		
Isophorone	78-59-1	2	1	0.29	ug/L	51	110	20	51	110	20		
Naphthalene	91-20-3	1	0.5	0.3	ug/L	39	110	20	39	110	20		
Nitrobenzene	98-95-3	1	0.5	0.45	ug/L	52	110	20	52	110	20		
N-Nitrosodiethylamine	55-18-5	5	2.5	1.14	ug/L	53	110	20	53	110	20		
N-Nitrosodimethylamine	62-75-9	10	5	1.37	ug/L	27	100	20	27	100	20		
N-Nitrosodi-n-butylamine	924-16-3	5	2.5	0.98	ug/L	51	116	20	51	116	20		
N-Nitrosodi-n-propylamine	621-64-7	0.5	0.25	0.14	ug/L	43	110	20	43	110	20		

N-Nitrosodiphenylamine	86-30-6	1	0.5	0.34	ug/L	64	110	20	64	110	20		
N-Nitrosomethylethylamine	10595-95-6	5	2.5	1.09	ug/L	42	110	20	42	110	20		
N-Nitrosopiperidine	100-75-4	5	2.5	0.81	ug/L	59	110	20	59	110	20		
N-Nitrosopyrrolidine	930-55-2	5	2.5	0.79	ug/L	37	116	20	37	116	20		
Pentachlorobenzene	608-93-5	5	2.5	1.07	ug/L	55	110	20	55	110	20		
Pentachloroethane	76-01-7	10	5	1.77	ug/L	50	150	20	50	150	20		
Pentachlorophenol	87-86-5	20	10	5.6	ug/L	42	127	20	42	127	20		
Phenanthrene	85-01-8	1	0.5	0.35	ug/L	59	110	20	59	110	20		
Phenol	108-95-2	5	2.5	0.36	ug/L	20	100	20	20	100	20		
Pyrene	129-00-0	1	0.5	0.48	ug/L	58	115	20	58	115	20		
Pyridine	110-86-1	20	10	7.2	ug/L	10	100	20	10	100	20		
2,3,4,6-Tetrachlorophenol	58-90-2	5	2.5	1.51	ug/L	60	112	20	60	112	20		
2-sec-Butyl-4,6-dinitrophenol	88-85-7	10	5	3.24	ug/L	40	110	20	40	110	20		
3,3'-Dimethylbenzidine	119-93-7	20	10	9.09	ug/L	10	110	20	10	110	20		
Diphenylamine	122-39-4	5	2.5	1.73	ug/L	50	150		50	150			
2-Fluorophenol (Surr)	367-12-4				ug/L							20	100
Phenol-d5 (Surr)	4165-62-2				ug/L							20	100
Nitrobenzene-d5 (Surr)	4165-60-0				ug/L							41	110
2-Fluorobiphenyl	321-60-8				ug/L							48	110
2,4,6-Tribromophenol (Surr)	118-79-6				ug/L							50	129
Terphenyl-d14 (Surr)	1718-51-0				ug/L							44	132

MDL W GCMS BNA 8270D_ICAL 1	Liquid-Liquid Extraction (Separatory Funnel)	3510C

MDL W GCMS BNA 8270D_APIX	Semivolatile Organic Compounds (GC/MS)	8270D / C

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,4-Dinitrobenzene	100-25-4	5	2.5	2.01	ug/L								
1,4-Dioxane	123-91-1	20	10	6.91	ug/L	10	100	20	10	100	20		
1,4-Naphthoquinone	130-15-4	10	5	1.73	ug/L								
1-Naphthylamine	134-32-7	10	5	1.41	ug/L								
2,3,4,6-Tetrachlorophenol	58-90-2	5	2.5	1.51	ug/L	60	112	20	60	112	20		
2,3,5,6-Tetrachlorophenol	935-95-5	5	2.5	2.95	ug/L								
2-Acetylaminofluorene	53-96-3	5	2.5	0.98	ug/L								
2-Naphthylamine	91-59-8	10	5	1.44	ug/L								
2-Picoline	109-06-8	10	5	1.26	ug/L								
2-sec-Butyl-4,6-dinitrophenol	88-85-7	10	5	3.24	ug/L	40	110	20	40	110	20		
3,3'-Dimethylbenzidine	119-93-7	20	10	9.09	ug/L	10	110	20	10	110	20		
3-Methylcholanthrene	56-49-5	5	2.5	0.98	ug/L								
4-Aminobiphenyl	92-67-1	10	5	1.26	ug/L								
4-Nitroquinoline-1-oxide	56-57-5	20	12	11.8	ug/L								
5-Nitro-o-toluidine	99-55-8	5	2.5	1.55	ug/L								
7,12-Dimethylbenz(a)anthracene	57-97-6	5	2.5	2.21	ug/L								
alpha,alpha-Dimethyl phenethylamine	122-09-8	40	20	8.58	ug/L								
Aramite	140-57-8	5	2.5	1.29	ug/L								
Chlorobenzilate	510-15-6	5	2.5	1.35	ug/L								
Diallate	2303-16-4	5	2.5	2.23	ug/L								
Dibenz[a,j]acridine	224-42-0	5	2.5	1.29	ug/L								
Diphenylamine	122-39-4	5	2.5	1.73	ug/L	50	150		50	150			
Ethyl methanesulfonate	62-50-0	5	2.5	1.95	ug/L								
Hexachlorophene	70-30-4	100	52	52	ug/L								
Isodrin	465-73-6	5	2.5	2.3	ug/L								
Isosafrole	120-58-1	5	2.5	1.75	ug/L								
Kepone	143-50-0	10	5	1.34	ug/L								
m-Dinitrobenzene	99-65-0	5	2.5	1.93	ug/L								
Methapyrilene	91-80-5	40	20	6.51	ug/L								
Methyl methanesulfonate	66-27-3	5	2.5	1.83	ug/L								
N-Nitrosomorpholine	59-89-2	5	2.5	2.42	ug/L								
o-Toluidine	95-53-4	5	2.5	1.64	ug/L								
p-Dimethylamino azobenzene	60-11-7	5	2.5	1.25	ug/L								
Pentachloronitrobenzene	82-68-8	5	2.5	1.69	ug/L								

Phenacetin	62-44-2	5	2.5	1.82	ug/L								
p-Phenylene diamine	106-50-3	40	20	20	ug/L								
Pronamide	23950-58-5	10	5	1.1	ug/L								
Safrole, Total	94-59-7	5	2.5	1.86	ug/L								
sym-Trinitrobenzene	99-35-4	5	2.5	2.3	ug/L								
2-Fluorophenol (Surr)	367-12-4				ug/L							20	100
Phenol-d5 (Surr)	4165-62-2				ug/L							20	100
Nitrobenzene-d5 (Surr)	4165-60-0				ug/L							41	110
2-Fluorobiphenyl	321-60-8				ug/L							48	110
2,4,6-Tribromophenol (Surr)	118-79-6				ug/L							50	129
Terphenyl-d14 (Surr)	1718-51-0				ug/L							44	132

MDL W GCMS BNA 8270D_APIX	Liquid-Liquid Extraction (Separatory Funnel	3510C

MDL W GCMS BNA 8270D_Pest	Semivolatile Organic Compounds (GC/MS)	8270D / C

Analyte Description	CAS Number	RL Limit	LOD Limit	MDL Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
4,4'-Stilbenediol,a,a'-diethyl	56-53-1	20	10	2.11	ug/L								
Aldicarb	116-06-3	20	10	9.13	ug/L								
Benzyl benzoate	120-51-4	10	5	1.5	ug/L								
Carbofuran	1563-66-2	10	5	1.37	ug/L	50	150	20	50	150	20		
Dimethoate	60-51-5	10	5	1.5	ug/L								
Disulfoton	298-04-4	10	5	1.37	ug/L								
Ethyl Parathion	56-38-2	10	5	1.66	ug/L								
Famphur	52-85-7	10	5	1.49	ug/L								
Malathion	121-75-5	10	5	1.65	ug/L								
Methyl parathion	298-00-0	10	5	1.61	ug/L								
o,o',o"-Triethylphosphorothioate	126-68-1	10	5	1.48	ug/L								
Phorate	298-02-2	10	5	1.48	ug/L								
Sulfotepp	3689-24-5	10	5	1.12	ug/L								
Thionazin	297-97-2	10	5	1.82	ug/L								
2-Fluorophenol (Surr)	367-12-4				ug/L							20	100
Phenol-d5 (Surr)	4165-62-2				ug/L							20	100
Nitrobenzene-d5 (Surr)	4165-60-0				ug/L							41	110
2-Fluorobiphenyl	321-60-8				ug/L							48	110
2,4,6-Tribromophenol (Surr)	118-79-6				ug/L							50	129
Terphenyl-d14 (Surr)	1718-51-0				ug/L							44	132

MDL W GCMS BNA 8270D_Pest	Liquid-Liquid Extraction (Separatory Funnel)	3510C

MDL S GCMS BNA 8270D_ICAL1	Semivolatile Organic Compounds (GC/MS)	8270D / C

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,2,4,5-Tetrachlorobenzene	95-94-3	167	83	39.1	ug/Kg	63	110	30	63	110	30		
1,2,4-Trichlorobenzene	120-82-1	167	83	37.6	ug/Kg	57	110	30	57	110	30		
1,2-Dichlorobenzene	95-50-1	167	83	36.3	ug/Kg	55	110	30	55	110	30		
1,2-Diphenylhydrazine	122-66-7	167	83	42.1	ug/Kg	53	112	30	53	112	30		
1,3-Dichlorobenzene	541-73-1	167	83	34.9	ug/Kg	52	110	30	52	110	30		
1,4-Dichlorobenzene	106-46-7	167	83	34.9	ug/Kg	52	110	30	52	110	30		
2,2'-oxybis[1-chloropropane]	108-60-1	167	83	36.8	ug/Kg	36	110	30	36	110	30		
2,4,5-Trichlorophenol	95-95-4	330	167	95	ug/Kg	57	113	30	57	113	30		
2,4,6-Trichlorophenol	88-06-2	330	167	41.7	ug/Kg	55	112	30	55	112	30		
2,4-Dichlorophenol	120-83-2	330	167	101	ug/Kg	60	110	30	60	110	30		
2,4-Dimethylphenol	105-67-9	330	167	104	ug/Kg	52	110	30	52	110	30		
2,4-Dinitrophenol	51-28-5	670	330	170	ug/Kg	10	110	30	10	110	30		
2,4-Dinitrotoluene	121-14-2	167	83	50.9	ug/Kg	57	116	30	57	116	30		
2,6-Dichlorophenol	87-65-0	167	83	47.2	ug/Kg	63	110	30	63	110	30		
2,6-Dinitrotoluene	606-20-2	167	83	39.5	ug/Kg	60	110	30	60	110	30		
2-Chloronaphthalene	91-58-7	167	83	37.4	ug/Kg	54	110	30	54	110	30		
2-Chlorophenol	95-57-8	167	83	47.5	ug/Kg	53	110	30	53	110	30		
2-Methylnaphthalene	91-57-6	167	83	43.1	ug/Kg	51	110	30	51	110	30		
2-Methylphenol	95-48-7	167	83	44.1	ug/Kg	48	110	30	48	110	30		
2-Nitroaniline	88-74-4	167	83	59.8	ug/Kg	53	126	30	53	126	30		
2-Nitrophenol	88-75-5	330	167	52.1	ug/Kg	54	112	30	54	112	30		
3 & 4 Methylphenol	15831-10-4	167	83	62.9	ug/Kg	44	121	30	44	121	30		
3,3'-Dichlorobenzidine	91-94-1	167	83	27.7	ug/Kg	31	110	30	31	110	30		
3-Nitroaniline	99-09-2	330	167	64.1	ug/Kg	36	110	30	36	110	30		
4,6-Dinitro-2-methylphenol	534-52-1	330	167	80.6	ug/Kg	10	110	30	10	110	30		
4-Bromophenyl phenyl ether	101-55-3	167	83	37.1	ug/Kg	58	111	30	58	111	30		
4-Chloro-3-methylphenol	59-50-7	330	167	159	ug/Kg	54	111	30	54	111	30		
4-Chloroaniline	106-47-8	670	330	101	ug/Kg	25	110	30	25	110	30		
4-Chlorophenyl phenyl ether	7005-72-3	167	83	52.3	ug/Kg	57	110	30	57	110	30		
4-Nitroaniline	100-01-6	330	167	68.1	ug/Kg	44	124	30	44	124	30		
4-Nitrophenol	100-02-7	670	330	179	ug/Kg	39	125	30	39	125	30		
Acenaphthene	83-32-9	33	16.7	9.93	ug/Kg	53	110	30	53	110	30		
Acenaphthylene	208-96-8	33	16.7	7.63	ug/Kg	51	110	30	51	110	30		
Acetophenone	98-86-2	330	167	60	ug/Kg	35	100	30	35	100	30		

Aniline	62-53-3	670	330	300	ug/Kg	34	100	30	34	100	30		
Anthracene	120-12-7	33	16.7	7.81	ug/Kg	52	110	30	52	110	30		
Benzidine	92-87-5	670	330	330	ug/Kg	10	100	30	10	100	30		
Benzo[a]anthracene	56-55-3	33	16.7	6.96	ug/Kg	57	110	30	57	110	30		
Benzo[a]pyrene	50-32-8	33	16.7	6.05	ug/Kg	56	110	30	56	110	30		
Benzo[b]fluoranthene	205-99-2	33	16.7	6.45	ug/Kg	50	110	30	50	110	30		
Benzo[g,h,i]perylene	191-24-2	33	16.7	11.2	ug/Kg	54	117	30	54	117	30		
Benzo[k]fluoranthene	207-08-9	33	16.7	7.92	ug/Kg	43	121	30	43	121	30		
Benzoic acid	65-85-0	1670	830	457	ug/Kg	10	100	30	10	100	30		
Benzyl alcohol	100-51-6	330	167	99.3	ug/Kg	44	110	30	44	110	30		
Bis(2-chloroethoxy)methane	111-91-1	167	83	36.7	ug/Kg	56	110	30	56	110	30		
Bis(2-chloroethyl)ether	111-44-4	167	83	49.2	ug/Kg	48	110	30	48	110	30		
Bis(2-ethylhexyl) phthalate	117-81-7	167	83	44	ug/Kg	56	114	30	56	114	30		
Butyl benzyl phthalate	85-68-7	167	83	41.6	ug/Kg	60	120	30	60	120	30		
Carbazole	86-74-8	167	83	46.7	ug/Kg	57	110	30	57	110	30		
Chrysene	218-01-9	33	16.7	7.5	ug/Kg	54	110	30	54	110	30		
Dibenz(a,h)anthracene	53-70-3	33	16.7	9.28	ug/Kg	52	118	30	52	118	30		
Dibenzofuran	132-64-9	167	83	39.9	ug/Kg	54	110	30	54	110	30		
Diethyl phthalate	84-66-2	167	83	55.4	ug/Kg	58	112	30	58	112	30		
Dimethyl phthalate	131-11-3	167	83	41.5	ug/Kg	60	110	30	60	110	30		
Di-n-butyl phthalate	84-74-2	167	83	41.9	ug/Kg	56	117	30	56	117	30		
Di-n-octyl phthalate	117-84-0	167	83	67.4	ug/Kg	49	121	30	49	121	30		
Fluoranthene	206-44-0	33	16.7	13.6	ug/Kg	55	113	30	55	113	30		
Fluorene	86-73-7	33	16.7	7.55	ug/Kg	52	112	30	52	112	30		
Hexachlorobenzene	118-74-1	67	33	6.54	ug/Kg	54	114	30	54	114	30		
Hexachlorobutadiene	87-68-3	167	83	43.5	ug/Kg	53	110	30	53	110	30		
Hexachlorocyclopentadiene	77-47-4	670	330	154	ug/Kg	10	112	30	10	112	30		
Hexachloroethane	67-72-1	167	83	35.4	ug/Kg	51	110	30	51	110	30		
Hexachloropropene	1888-71-7	330	83	132	ug/Kg	54	110	30	54	110	30		
Indeno[1,2,3-cd]pyrene	193-39-5	33	16.7	11.2	ug/Kg	53	116	30	53	116	30		
Isophorone	78-59-1	167	83	37	ug/Kg	49	110	30	49	110	30		
Naphthalene	91-20-3	33	16.7	6.4	ug/Kg	48	110	30	48	110	30		
Nitrobenzene	98-95-3	33	16.7	10.3	ug/Kg	52	110	30	52	110	30		
N-Nitrosodiethylamine	55-18-5	330	167	95.3	ug/Kg	50	110	30	50	110	30		
N-Nitrosodimethylamine	62-75-9	670	363	363	ug/Kg	42	110	30	42	110	30		
N-Nitrosodi-n-butylamine	924-16-3	167	83	59.5	ug/Kg	46	138	30	46	138	30		
N-Nitrosodi-n-propylamine	621-64-7	167	83	42.2	ug/Kg	40	121	30	40	121	30		

N-Nitrosodiphenylamine	86-30-6	167	83	44.9	ug/Kg	58	110	30	58	110	30		
N-Nitrosomethylethylamine	10595-95-6	670	330	272	ug/Kg	49	114	30	49	114	30		
N-Nitrosopiperidine	100-75-4	330	167	30.6	ug/Kg	56	123	30	56	123	30		
N-Nitrosopyrrolidine	930-55-2	167	83	38.1	ug/Kg	46	116	30	46	116	30		
Pentachlorobenzene	608-93-5	167	83	41.9	ug/Kg	70	110	30	70	110	30		
Pentachloroethane	76-01-7	167	83	36.2	ug/Kg	50	150	30	50	150	30		
Pentachlorophenol	87-86-5	670	330	169	ug/Kg	20	117	30	20	117	30		
Phenanthrene	85-01-8	33	16.7	13.9	ug/Kg	51	116	30	51	116	30		
Phenol	108-95-2	167	83	52.6	ug/Kg	49	110	30	49	110	30		
Pyrene	129-00-0	33	16.7	12	ug/Kg	50	112	30	50	112	30		
Pyridine	110-86-1	670	330	394	ug/Kg	24	100	30	24	100	30		
2,3,4,6-Tetrachlorophenol	58-90-2	167	83	45.4	ug/Kg	45	116	30	45	116	30		
2-sec-Butyl-4,6-dinitrophenol	88-85-7	330	167	85.7	ug/Kg	50	150	30	50	150	30		
3,3'-Dimethylbenzidine	119-93-7	670	330	173	ug/Kg	10	100	30	10	100	30		
Diphenylamine	122-39-4	167	83	38.2	ug/Kg	50	150		50	150			
2-Fluorophenol (Surr)	367-12-4				ug/Kg							30	110
Phenol-d5 (Surr)	4165-62-2				ug/Kg							31	110
Nitrobenzene-d5 (Surr)	4165-60-0				ug/Kg							30	115
2-Fluorobiphenyl	321-60-8				ug/Kg							30	119
2,4,6-Tribromophenol (Surr)	118-79-6				ug/Kg							35	137
Terphenyl-d14 (Surr)	1718-51-0				ug/Kg							36	134

MDL S GCMS BNA 8270D_ICAL1	Automated Soxhlet Extraction	3541

MDL S GCMS BNA 8270D_APIX	Semivolatile Organic Compounds (GC/MS)	8270D / C

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,4-Dichlorobenzene	106-46-7	167	83	34.9	ug/Kg	52	110	30	52	110	30		
1,4-Dioxane	123-91-1	670	330	225	ug/Kg	10	100	30	10	100	30		
1,4-Naphthoquinone	130-15-4	670	330	330	ug/Kg								
1-Naphthylamine	134-32-7	167	83	25.5	ug/Kg								
2,3,4,6-Tetrachlorophenol	58-90-2	167	83	45.4	ug/Kg	45	116	30	45	116	30		
2,3,5,6-Tetrachlorophenol	935-95-5	167	83	140	ug/Kg								
2-Acetylaminofluorene	53-96-3	167	83	30.8	ug/Kg								
2-Naphthylamine	91-59-8	167	83	45.2	ug/Kg								
2-Picoline	109-06-8	330	167	125	ug/Kg								
2-sec-Butyl-4,6-dinitrophenol	88-85-7	330	167	85.7	ug/Kg	50	150	30	50	150	30		
3,3'-Dimethylbenzidine	119-93-7	670	330	173	ug/Kg	10	100	30	10	100	30		
3-Methylcholanthrene	56-49-5	167	83	14	ug/Kg								
4-Aminobiphenyl	92-67-1	167	83	67.3	ug/Kg								
4-Nitroquinoline-1-oxide	56-57-5	670	330	314	ug/Kg								
5-Nitro-o-toluidine	99-55-8	167	83	31.4	ug/Kg								
7,12-Dimethylbenz(a)anthracene	57-97-6	167	83	40.6	ug/Kg								
alpha,alpha-Dimethyl phenethylamine	122-09-8	1330	670	394	ug/Kg								
Aramite	140-57-8	167	83	33	ug/Kg								
Chlorobenzilate	510-15-6	167	83	20.9	ug/Kg								
Diallate	2303-16-4	167	83	33.2	ug/Kg								
Dibenz[a,j]acridine	224-42-0	167	83	18.3	ug/Kg								
Diphenylamine	122-39-4	167	83	38.2	ug/Kg	50	150		50	150			
Ethyl methanesulfonate	62-50-0	167	83	18.8	ug/Kg								
Hexachlorophene	70-30-4	3300	2100	1230	ug/Kg								
Isodrin	465-73-6	167	83	49.4	ug/Kg								
Isosafrole	120-58-1	167	83	17.9	ug/Kg								
Kepone	143-50-0	670	330	330	ug/Kg								
m-Dinitrobenzene	99-65-0	167	83	31.3	ug/Kg								
Methapyrilene	91-80-5	1330	670	173	ug/Kg								
Methyl methanesulfonate	66-27-3	167	83	27	ug/Kg								
N-Nitrosomorpholine	59-89-2	167	83	30.9	ug/Kg								
o-Toluidine	95-53-4	167	83	32.2	ug/Kg								
p-Dimethylamino azobenzene	60-11-7	167	83	17.8	ug/Kg								
Pentachloronitrobenzene	82-68-8	167	83	23.6	ug/Kg								

Phenacetin	62-44-2	167	83	34.5	ug/Kg									
p-Phenylene diamine	106-50-3	1330	670	70	ug/Kg									
Pronamide	23950-58-5	167	83	26.9	ug/Kg									
Safrole	94-59-7	167	83	15.8	ug/Kg									
sym-Trinitrobenzene	99-35-4	670	340	339	ug/Kg									
2-Fluorophenol (Surr)	367-12-4				ug/Kg							30	110	
Phenol-d5 (Surr)	4165-62-2				ug/Kg							31	110	
Nitrobenzene-d5 (Surr)	4165-60-0				ug/Kg							30	115	
2-Fluorobiphenyl	321-60-8				ug/Kg							30	119	
2,4,6-Tribromophenol (Surr)	118-79-6				ug/Kg							35	137	
Terphenyl-d14 (Surr)	1718-51-0				ug/Kg							36	134	

MDL S GCMS BNA 8270D_APIX	Automated Soxhlet Extraction	3541

MDL S GCMS BNA 8270D_Pest	Semivolatile Organic Compounds (GC/MS)	8270D / C

Analyte Description	CAS Number	RL Limit	LOD Limit	MDL Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
4,4'-Stilbenediol,a,a'-diethyl	56-53-1	670	330	82.8	ug/Kg								
Aldicarb	116-06-3	670	330	366	ug/Kg								
Benzyl benzoate	120-51-4	330	167	50.3	ug/Kg								
Carbofuran	1563-66-2	330	167	76.6	ug/Kg	50	150	30	50	150	30		
Dimethoate	60-51-5	330	167	74.6	ug/Kg								
Disulfoton	298-04-4	330	167	50.2	ug/Kg								
Ethyl Parathion	56-38-2	330	167	92.6	ug/Kg								
Famphur	52-85-7	330	167	53.9	ug/Kg								
Malathion	121-75-5	330	167	98.9	ug/Kg								
Methyl parathion	298-00-0	330	167	93.7	ug/Kg								
o,o',o"-Triethylphosphorothioate	126-68-1	330	167	51.1	ug/Kg								
Phorate	298-02-2	330	167	70.2	ug/Kg								
Sulfotepp	3689-24-5	330	167	66.4	ug/Kg								
Thionazin	297-97-2	330	167	74	ug/Kg								
2-Fluorophenol (Surr)	367-12-4				ug/Kg							30	110
Phenol-d5 (Surr)	4165-62-2				ug/Kg							31	110
Nitrobenzene-d5 (Surr)	4165-60-0				ug/Kg							30	115
2-Fluorobiphenyl	321-60-8				ug/Kg							30	119
2,4,6-Tribromophenol (Surr)	118-79-6				ug/Kg							35	137
Terphenyl-d14 (Surr)	1718-51-0				ug/Kg							36	134

MDL S GCMS BNA 8270D_Pest	Automated Soxhlet Extraction	3541

Table 3.

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	10 - 80% of Base Peak
68	<2% of mass 69
70	<2% of mass 69
127	10 - 80% of Base Peak
197	<2% of mass 198
198	Base peak, or > 50% of Mass 442
199	5 - 9% of mass 198
275	10 - 60% of Base Peak
365	>1% of mass 198
441	Present but < 24% of mass 442
442	Base Peak, or > 50% of mass 198
443	15 - 24% of mass 442

Table 4

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Table 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010

Semivolatile Compounds	Minimum Response Factor (RF)
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 5.

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation.

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	a,a-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d8 (surr)	1-Naphthylamine
N-Nitroso-di-n-propylamine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetrachlorobenzene
		2,3,4,6-Tetrachlorophenol
		2,4,6-Tribromophenol (surr)
		2,4,6-Trichlorophenol
		2,4,5-Trichlorophenol

(surr) = Surrogate

Table 5. (cont.)

Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation.

Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'Dichlorobenzidine	Dibenz(a,h)anthracene
Fluoranthene	p-Dimethyl aminoazobenzene	7,12-Dimethylbenz(a)anthracene
Hexachlorobenzene	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Terphenyl-d14 (surr)	Indeno(1,2,3-cd) pyrene
Pentachlorophenol		3-Methylcholanthrene
Pentachloronitrobenzene		
Phenacetin		
Phenanthrene		
Pronamide		

(Surr) = Surrogate

Attachment 1.

Example: BNA Method for Standards and Samples

1. GC Oven Parameters

Initial Temp.	40°C
Initial Time	1.0 min.

Ramp Rate (°C / min.)	Final Temp. (°C)	Final Time (min.)
22	220	0
35	325	4.5

2. GC Electronic Pressure Control (EPC) Parameters

Constant Flow:	2.0 mL / minute

3. Mass Spectrometer Parameters

Scan Range:	35 - 500 m/z
Threshold:	150
A/D Ratio:	4
Scan/second:	2
Solvent Delay (scan start time):	Before the elution of the first compound
Run Time (scan stop time):	Until after the elution of last compound

Attachment 2.

Example: CA-T-P-002:
TestAmerica Corporate Policy for the Selection of Calibration Points
(050-001 to 050-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

4/13/2011	4/1/2011
Richard Burrows — Date	Dr. Charles W. Carter — Date
Director of Technical Services	VP, Quality & Technical Services

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(050-001)

1.0 PURPOSE

This policy describes TestAmerica's requirements for determination of the number of points, and removal of points from calibration curves.

2.0 SCOPE

This Policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP) that is what will be used.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the Data Quality Objectives (DQOs) for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a simple point at the reporting level.

Examples:

A. Need to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by UV-HPLC at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the

recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the data quality objectives will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in laboratory for meeting regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A five-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the Quality Assurance (QA) Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24 hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internals show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (all analytes in spike mix must

show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers) the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.

Attachment 3.

Example: Initial Calibration - Form 6
(051-001 to 051-005)

TestAmerica Laboratories
Initial Calibration Report

Method: \\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m
Instrument: CMS01 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Compound Type
Integrator: RTE Last Modified: 28-Nov-2012 16:54:03
No.Compounds:179

Initial Calibration Batches

Ical Batch: \\chi-svr07\chromdata\CMS01\20101230-1900.b
Inj Date : 30-Dec-2010 12:11:30, Sublist: chrom-1-8270*sub44

Ical Batch: \\chi-svr07\chromdata\CMS01\20111028-6722.b
Inj Date : 28-Oct-2011 14:03:30, Sublist: chrom-1-8270*sub45

Ical Batch: \\chi-svr07\chromdata\CMS01\20120109-8003.b
Inj Date : 09-Jan-2012 12:00:30, Sublist: chrom-1-8270*sub13

Ical Batch: \\chi-svr07\chromdata\CMS01\20120704-11261.b
Inj Date : 04-Jul-2012 18:35:30, Sublist: chrom-1-8270*sub29

Ical Batch: \\chi-svr07\chromdata\CMS01\20121128-14067.b
Inj Date : 28-Nov-2012 11:35:30, Sublist: chrom-1-8270*sub16

Limit Group: MSBNA_8270D_ICAL

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	Level 11	b	M1	M2	%RSD/R^2
* 1 1,4-Dichlorobenzene-d4	115935	133324	151505	97162	131672	93781	116941	105934	132123	73571	69909				
* 2 Naphthalene-d8	415731	486914	558063	388638	547564	430625	508423	498801	569373	405282	435605				
* 3 Acenaphthene-d10	200820	242948	303634	249186	303429	272155	300053	318018	346067	275040	335452				
* 4 Phenanthrene-d10	337450	412978	540635	482096	602678	529907	596374	620126	655635	514259	571077				
* 5 Chrysene-d12	289968	359394	570893	461837	598070	503743	549400	567006	666199	464050	455974				
* 6 Perylene-d12	282268	334314	519342	406793	538884	418595	472006	471557	569193	384492	302431				
$ 7 2-Fluorophenol				1.416340	1.314161	1.230433	1.289098	1.321379	1.328915	1.406514	1.357955		1.333099		Avg 4.6
$ 8 Phenol-d5				1.820559	1.677447	1.590168	1.687662	1.718178	1.740886	1.832205	1.687935		1.719380		Avg 4.6
$ 9 Nitrobenzene-d5				0.477226	0.419341	0.407159	0.411850	0.439816	0.434201	0.439884	0.441502		0.433872		Avg 5.1
$ 10 2-Fluorobiphenyl				1.495334	1.324700	1.331577	1.259724	1.357137	1.341076	1.401461	1.381656		1.361583		Avg 5.0
$ 11 2,4,6-Tribromophenol				0.172238	0.165930	0.169848	0.194088	0.200748	0.222225	0.229809	0.209542		0.195553		Avg 13
$ 12 Terphenyl-d14				0.867008	0.770244	0.772721	0.692920	0.783165	0.710201	0.726509	0.857539		0.772538		Avg 8.3
13 N-Nitrosodimethylamine					Disabled	0.336944	0.644951	0.619576	0.706406	0.700409	0.750642	-4.09512	0.777861		Linr 0.995
14 Pyridine						0.728397	1.428544	1.474387	1.495861	1.613217	1.548371	-8.39706	1.691854		Linr 0.997
15 1,4-Dioxane					0.327910	0.333903	0.463893	0.449675	0.466927	0.429334	0.444078		0.416531		Avg 14
16 2-Picoline					1.501652	1.543427	1.582885	1.544382	1.593077	1.706820	1.649753		1.588857		Avg 4.4
17 N-Nitrosomethylethylam					0.611318	0.623050	0.516812	0.627033	0.510458	0.682096	0.644449		0.602174		Avg 11

(100-190)

Report Date: 01-Dec-2012 13:09:48 Chrom Revision: 2.0 22-Oct-2012 16:30:48 Page: 2

Method: \\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	Level 11	b	M1	M2	%RSD/R^2
19 Methyl methanesulfonat					0.846832	0.849246	0.879044	0.855993	0.876274	0.926984	0.873613		0.872569		Avg 3.1
21 N-Nitrosodiethylamine					0.625921	0.580406	0.612844	0.611702	0.625685	0.673020	0.625288		0.622124		Avg 4.4
23 Aldicarb				0.050555	0.057513	0.053529	0.051982	0.071469	0.055736	0.074416	0.096576		0.063972		Avg *25 R1
22 Ethyl methanesulfonate					1.170852	1.166017	1.205941	1.193340	1.225971	1.302773	1.208381		1.210468		Avg 3.8
24 Phenol					1.820877	1.853917	1.969654	1.927302	1.984416	2.019104	1.949289		1.932080		Avg 3.7
26 Aniline					2.083523	2.094961	2.183893	2.206498	2.268254	2.369577	2.239351		2.206580		Avg 4.5
28 Pentachloroethane					0.602135	0.607635	0.620156	0.623840	0.630376	0.674181	0.654565		0.630413		Avg 4.1
27 Bis(2-chloroethyl)ethe				1.592596	1.335478	1.337127	1.395207	1.374490	1.398668	1.555399	1.372692		1.420207		Avg 6.9
25 Benzaldehyde						0.848190	0.897400	0.897904	0.915472	0.873051	0.886085		0.886351		Avg 2.6
29 2-Chlorophenol				1.626392	1.353713	1.416422	1.536665	1.519597	1.555894	1.638473	1.537048		1.523025		Avg 6.4
30 1,3-Dichlorobenzene				1.771757	1.556794	1.491670	1.607301	1.575202	1.589040	1.719797	1.634813		1.618297		Avg 5.6
31 1,4-Dichlorobenzene				1.781486	1.505920	1.534288	1.597587	1.587582	1.623003	1.728119	1.657823		1.626976		Avg 5.7
32 Benzyl alcohol					0.880997	0.837904	0.935005	0.931696	0.965378	1.021108	0.942749		0.930691		Avg 6.3
33 1,2-Dichlorobenzene				1.741186	1.495969	1.456453	1.528801	1.542702	1.572323	1.651703	1.586338		1.571934		Avg 5.7
34 2-Methylphenol				1.304184	1.009407	1.055956	1.126231	1.100817	1.146126	1.207001	1.080811		1.128817		Avg 8.2
35 2,2'-oxybis[1-chloropr				2.115527	1.808984	1.760025	1.864336	1.796930	1.797944	1.903143	1.795288		1.855272		Avg 6.2
40 N-Nitrosopyrrolidine					0.677794	0.643135	0.735357	0.712935	0.772448	0.801329	0.648021		0.713003		Avg 8.5
37 3 & 4 Methylphenol			1.573127	1.390793	1.281208	1.262190	1.371606	1.254296	1.294114	1.367901	1.146894		1.326903		Avg 9.0
38 N-Nitrosodi-n-propylam		1.473463	1.332134	1.229422	1.096015	1.082379	1.155262	1.101554	1.123567	1.166599	0.997322		1.175772		Avg 12
39 Acetophenone					1.978603	1.916702	2.067926	1.912889	1.935713	1.999408	1.793861		1.943586		Avg 4.4
43 Phenylmercaptan			0.841040	0.932210		1.241967	1.253043	1.156771	1.070187	0.837401	0.682282	-0.63082	1.408812	-0.00795	Quad 0.993
41 2-Toluidine					2.088065	2.054628	2.317333	2.328371	2.431788	2.474771	2.202209		2.271024		Avg 7.1
42 Hexachloroethane					0.734506	0.723569	0.775385	0.770980	0.787990	0.839942	0.794931		0.775329		Avg 5.0
44 Nitrobenzene			0.452096	0.487494	0.424682	0.412602	0.413909	0.423228	0.420894	0.427681	0.425266		0.431983		Avg 5.5
62 Indene			1.794932	1.833031		1.808228	1.818304	1.606516	1.458387	1.140370	0.911659	-0.27785	2.053860	-0.01312	Quad 0.997
46 N-Nitrosopiperidine					0.172448	0.176572	0.174835	0.182796	0.182473	0.189811	0.183135		0.180296		Avg 3.3
47 Isophorone				0.858342	0.739145	0.733166	0.720834	0.748250	0.729379	0.745702	0.739540		0.751795		Avg 5.8
73 p-Phenylene diamine							0.278723	0.330987	0.422925	0.368641	0.357727		0.351800		Avg 15
51 N-Nitrosomorpholine					0.578927	0.538479	0.552844	0.515946	0.558403	0.468802	0.591741		0.543592		Avg 7.6
48 2-Nitrophenol					0.191844	0.203403	0.208048	0.215872	0.215498	0.220703	0.215294		0.210095		Avg 4.7
49 2,4-Dimethylphenol						0.320061	0.323331	0.345632	0.342641	0.352663	0.322079		0.334401		Avg 4.2
50 Bis(2-chloroethoxy)met				0.521077	0.438553	0.446116	0.446739	0.464938	0.450422	0.460834	0.459274		0.460994		Avg 5.6
52 Benzoic acid						Disabled	0.131205	0.165772	0.200348	0.198581	0.151273		0.169436		Avg 18
110 o,o',o"-Triethylphosp				0.131711	0.135077	0.143345	0.153270	0.148699	0.144872	0.149469	0.149945		0.144548		Avg 5.2
54 2,4-Dichlorophenol					0.285427	0.280906	0.284746	0.304194	0.299096	0.307402	0.296549		0.294046		Avg 3.5
55 1,2,4-Trichlorobenzene				0.406059	0.332387	0.334678	0.339735	0.357235	0.348611	0.362672	0.358198		0.354947		Avg 6.6
56 Naphthalene			1.116625	1.181625	1.026892	1.016582	1.010328	1.042734	0.997613	1.004213	0.983085		1.042189		Avg 6.2
57 4-Chloroaniline					0.451465	0.440341	0.470630	0.482054	0.475402	0.476505	0.446501		0.463271		Avg 3.6
58 2,6-Dichlorophenol					0.280703	0.290211	0.303408	0.316381	0.311110	0.315093	0.307711		0.303517		Avg 4.4
59 Hexachloropropene					0.232146	0.238540	0.255751	0.273738	0.282352	0.282025	0.279003		0.263365		Avg 8.1
60 Hexachlorobutadiene					0.202778	0.205842	0.198208	0.215212	0.212141	0.221610	0.221332		0.211018		Avg 4.3
61 3,4-Dichlorophenol						0.639471	0.606673	0.623837	0.557788	0.533833	0.534250		0.582642		Avg 8.0
64 N-Nitrosodi-n-butylami					0.257886	0.267610	0.265147	0.275929	0.270792	0.275323	0.268430		0.268731		Avg 2.3
71 alpha,alpha-Dimethyl p					0.725364	0.392194	0.514775	0.331952	0.519932	0.472692	0.483799		0.491530		Avg *25 R1, RB

(051-003)

Report Date: 01-Dec-2012 13:09:48 Chrom Revision: 2.0 22-Oct-2012 16:30:48 Page: 3

Method: \\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	Level 11	b	M1	M2	%RSD/R^2
66 4-Chloro-3-methylpheno					0.310045	0.299274	0.311303	0.323796	0.328307	0.329263	0.298335		0.314332		Avg 4.1
65 Caprolactam						0.038616	0.061021	0.062156	0.067111	0.070859	0.073187	-0.37200	0.075909		Linr 0.998
76 Isosafrole					0.504876	0.491911	0.501346	0.503783	0.513381	0.535511	0.550567		0.514482		Avg 4.1
68 2-Methylnaphthalene		0.828927	0.750433	0.752707	0.664791	0.655920	0.664285	0.695791	0.683537	0.695952	0.657595		0.704994		Avg 7.9
69 Hexachlorocyclopentadi						0.085236	0.188217	0.275722	0.300201	0.337834	0.323324	-3.06402	0.365015		Linr 0.997
70 1,2,4,5-Tetrachloroben				0.708586	0.617251	0.612734	0.616367	0.662060	0.661823	0.685118	0.694027		0.657246		Avg 5.8
72 2,4,6-Trichlorophenol					0.374110	0.385250	0.393521	0.439261	0.439143	0.458731	0.448009		0.419718		Avg 8.2
74 2,4,5-Trichlorophenol					0.416428	0.370731	0.425195	0.460772	0.479095	0.496954	0.464047		0.444746		Avg 9.7
67 Safrole, Total					0.267218	0.253007	0.264844	0.274910	0.272547	0.283675	0.267461		0.269095		Avg 3.5
45 Quinoline			0.341663	0.450446		0.552392	0.592725	0.606441	0.558178	0.594066	0.488995	-0.17105	0.559927		Linr 0.992
77 2-Chloronaphthalene				1.518132	1.266181	1.196205	1.217890	1.282805	1.281134	1.281974	1.267933		1.289032		Avg 7.6
53 1-Methylnaphthalene			0.538471	0.545099		0.575216	0.606663	0.592561	0.544856	0.564834	0.477445		0.555643		Avg 7.2
79 2-Nitroaniline					0.397681	0.379289	0.409571	0.422292	0.435249	0.441454	0.412887		0.414060		Avg 5.2
78 1,1'-Biphenyl			1.300013		1.334819	1.351224	1.429762	1.334304	1.277552	1.062177	1.096946		1.273349		Avg 10
81 1,4-Naphthoquinone						0.430284	0.481809	0.508146	0.529344	0.542003	0.513240		0.500804		Avg 8.0
82 Dimethyl phthalate				1.598047	1.404260	1.379921	1.361025	1.430741	1.440448	1.474039	1.441277		1.441220		Avg 5.1
83 1,3-Dinitrobenzene				0.221892	0.221287	0.191157	0.215314	0.225636	0.235834	0.242112	0.226663		0.222487		Avg 6.8
84 2,6-Dinitrotoluene		0.315458	0.323087	0.372053	0.317725	0.315458	0.314990	0.328828	0.344021	0.346044	0.337531		0.331520		Avg 5.6
85 Acenaphthylene			2.255720	2.460879	2.028564	1.944298	1.984263	2.029203	2.041132	2.055444	1.955056		2.083840		Avg 8.1
86 3-Nitroaniline						0.313369	0.368223	0.379673	0.391619	0.407167	0.370285		0.371722		Avg 8.6
87 Acenaphthene			1.290070	1.358968	1.169274	1.145021	1.148760	1.214201	1.214351	1.228678	1.197206		1.218503		Avg 5.7
88 2,4-Dinitrophenol						Disabled	0.122396	0.152185	0.201078	0.186197	0.149968		0.162365		Avg 19
89 4-Nitrophenol						0.164668	0.223116	0.227685	0.249254	0.253293	0.210489		0.221417		Avg 15
90 Pentachlorobenzene					0.528407	0.513974	0.521174	0.562735	0.575523	0.583530	0.571657		0.551000		Avg 5.2
91 2,4-Dinitrotoluene			0.426674	0.489974	0.417524	0.397468	0.444373	0.450919	0.485039	0.493552	0.451095		0.450735		Avg 7.5
93 1,4-Dinitrobenzene					0.106600	0.119904	0.135323	0.136701	0.146930	0.154698	0.145300		0.135065		Avg 12
92 Dibenzofuran				2.033887	1.695145	1.610678	1.645606	1.728664	1.738646	1.723650	1.609803		1.723260		Avg 7.9
94 1-Naphthylamine					1.193273	0.995455	1.158203	1.286998	1.374747	1.390914	1.246346		1.235134		Avg 11
95 2,3,4,6-Tetrachlorophe					0.272651	0.260945	0.310253	0.339247	0.367972	0.377024	0.342065		0.324308		Avg 14
96 2-Naphthylamine					1.137922	0.782949	1.130928	1.211528	1.384063	1.306038	1.152289		1.157959		Avg 16
97 Diethyl phthalate				1.775190	1.591592	1.489676	1.500139	1.586080	1.585735	1.606422	1.594726		1.591195		Avg 5.5
98 4-Chlorophenyl phenyl					0.647942	0.641379	0.629343	0.670234	0.682223	0.690729	0.675777		0.662518		Avg 3.5
99 Fluorene			1.544543	1.625439	1.370898	1.322426	1.391950	1.449301	1.469571	1.475792	1.423527		1.452605		Avg 6.3
100 N-Nitro-o-toluidine					0.215061	0.212441	0.227934	0.236684	0.218938	0.236006	0.230662		0.225389		Avg 4.4
101 4-Nitroaniline						0.321557	0.379641	0.400971	0.357598	0.329353	0.339006		0.354688		Avg 8.7
102 4,6-Dinitro-2-methylph						0.100848	0.120905	0.135326	0.128137	0.149875	0.139507		0.129100		Avg 13
112 Thionazin				0.101216	0.113708	0.118145	0.125146	0.127117	0.121247	0.121145	0.129627		0.119669		Avg 7.5
103 N-Nitrosodiphenylamine			0.601120	0.587934	0.542822	0.532694	0.521433	0.566574	0.533961	0.572324	0.570600		0.558829		Avg 4.9
104 Diphenylamine			0.704212	0.688765	0.635916	0.624051	0.610859	0.663741	0.625535	0.670478	0.668458		0.654668		Avg 4.9
105 1,2-Diphenylhydrazine					1.732381	1.626509	1.634325	1.610233	1.666069	1.620242	1.625534		1.645042		Avg 2.6
106 2,3,5,6-Tetrachlorophe					0.268240	0.296417	0.313275	0.309163	0.328990	0.371644	0.322207		0.315705		Avg 10
108 Diallate					0.305218	0.316645	0.274844	0.296824	0.274434	0.299765	0.317425		0.297879		Avg 5.9
109 Phenacetin					0.404418	0.399125	0.396910	0.441214	0.409030	0.443837	0.403298		0.413976		Avg 4.8
107 1,3,5-Trinitrobenzene				0.206676	0.233824	0.220183	0.243220	0.268712	0.247543	0.310146	0.237054		0.245920		Avg 13
111 4-Bromophenyl phenyl e					0.211693	0.219419	0.206322	0.217007	0.213873	0.236336	0.231619		0.219467		Avg 4.9

(051-003)

Method: \\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	Level 11	b	M1	M2	%RSD/R^2
122 Pherate				0.405164	0.440442	0.424165	0.455809	0.439974	0.428126	0.418393	0.431547		0.430453		Avg 3.6
113 Hexachlorobenzene		0.362208	0.275201	0.258919	0.232017	0.240120	0.229030	0.257707	0.242792	0.268155	0.263432	0.018388	0.255747		Linr 0.997
126 Dimethoate				0.127950	0.215091	0.230837	0.268386	0.276826	0.274426	0.265181	0.263804	-0.28859	0.273565		Linr 0.999
125 Carbofuran				0.076765	0.102983	0.108324	0.136490	0.151310	0.158594	0.156769	0.162646	-0.24913	0.160441		Linr 0.997
115 4-Aminobiphenyl					0.719677	0.621066	0.706897	0.768297	0.721013	0.768489	0.715774		0.717316		Avg 6.9
116 Pentachlorophenol						Disabled	0.082371	0.099047	0.105158	0.120806	0.106739		0.102824		Avg 14
117 Pentachloronitrobenzen					0.112919	0.107736	0.107423	0.119351	0.114208	0.123346	0.123397		0.115483		Avg 5.8
114 Atrazine			Disabled		0.156987	0.171062	0.209683	0.232795	0.241761	0.238972	0.246214		0.213925		Avg 17
118 Pronamide					0.370353	0.357291	0.353598	0.373692	0.351705	0.374542	0.382083		0.366180		Avg 3.2
129 Benzyl benzoate				0.539071	0.641624	0.633593	0.689345	0.682227	0.678671	0.615008	0.635154		0.639337		Avg 7.6
121 Dinoseb					0.137683	0.148370	0.169883	0.197750	0.190980	0.203373	0.199103		0.178163		Avg 15
123 Phenanthrene			1.278118	1.223987	1.088880	1.078754	1.080231	1.200849	1.138732	1.188189	1.100817		1.153173		Avg 6.3
124 Anthracene			1.250454	1.236149	1.135816	1.125200	1.087850	1.167567	1.040246	1.110708	1.125159		1.142128		Avg 5.9
131 Disulfoton				0.310818	0.362457	0.352869	0.365323	0.359977	0.355282	0.340351	0.340361		0.348430		Avg 5.1
127 Carbazole					0.987968	0.963873	0.980454	1.019041	0.935222	1.012486	0.931126		0.975739		Avg 3.5
130 Di-n-butyl phthalate				1.642476	1.476444	1.433551	1.349814	1.387776	1.230992	1.289320	1.289132		1.387438		Avg 9.4
140 4-Nitroquinoline-1-oxi					0.016520	0.015660	0.015107	0.011503	0.009390	0.007569	0.008210		0.011994		Avg *31 R1, RB
136 Malathion				0.027073	0.035859	0.035925	0.038981	0.043296	0.044208	0.041382	0.043028		0.038719		Avg 15
135 Isodrin					0.167713	0.183297	0.152635	0.168567	0.143198	0.154990	0.244814		0.173602		Avg 20
134 Methyl parathion				0.130430	0.201851	0.211234	0.243443	0.251478	0.263725	0.247757	0.237138	-0.24810	0.251969		Linr 0.998
137 Fluoranthene			1.300414	1.253149	1.147683	1.065195	1.072083	1.171551	1.074470	1.119413	1.009952		1.134879		Avg 8.3
141 Methapyrilene					0.304400	0.272757	0.288567	0.256981	0.229571	0.213593	0.237704		0.257653		Avg 13
139 Benzidine						0.589451	0.706615	0.770755	0.754214	0.734304	0.749941		0.717546		Avg 9.3
142 Pyrene		1.689822	1.358605	1.396260	1.229497	1.285722	1.147353	1.219314	1.118909	1.121671	1.370389	0.206574	1.212740		Linr 0.992
138 Ethyl Parathion				0.098014	0.133851	0.130977	0.150738	0.155740	0.157828	0.143441	0.142937		0.139191		Avg 14
143 p-Dimethylamino azoben					0.196280	0.210050	0.201332	0.221177	0.205016	0.210405	0.233733		0.211142		Avg 6.0
144 Chlorobenzilate					0.337284	0.344742	0.305095	0.341482	0.312642	0.322558	0.370455		0.333465		Avg 6.6
146 Butyl benzyl phthalate				0.793763	0.722777	0.713437	0.650575	0.664786	0.582388	0.562510	0.672940		0.670397		Avg 11
147 3,3'-Dimethylbenzidine					0.738349	0.718026	0.734499	0.758861	0.730401	0.720632	0.734278		0.733578		Avg 1.8
149 Aramite, Total					0.046438	0.045437	0.049399	0.050951	0.045930	0.040849	0.053033		0.047434		Avg 8.5
150 Kepone						0.041471	0.054545	0.080298	0.077985	0.085775	0.076510	-0.50135	0.087963		Linr 0.992
159 Famphur				0.352687	0.416591	0.411472	0.450403	0.439052	0.433616	0.427375	0.395340		0.415817		Avg 7.4
158 Diethylstilbestrol				0.101430	0.128978	0.119810	0.149849	0.152568	0.135922	0.139679	0.134351		0.132823		Avg 12
153 2-Acetylaminofluorene					0.485077	0.481907	0.504529	0.516588	0.512947	0.517456	0.489457		0.501137		Avg 3.1
157 3,3'-Dichlorobenzidine					0.433602	0.419367	0.413922	0.442318	0.425131	0.423900	0.419758		0.425428		Avg 2.3
156 Bis(2-ethylhexyl) phth				1.176459	1.048077	1.057220	0.939199	0.969990	0.850264	0.805592	0.956334		0.975392		Avg 12
161 Benzo[a]anthracene	1.457264	1.580839	1.266515	1.374829	1.159656	1.136597	1.101751	1.215491	1.152891	1.193807	1.156422		1.254187		Avg 12
162 Chrysene	1.123986	1.467893	1.216913	1.281710	1.100082	1.052768	1.065755	1.091432	1.021202	0.996296	1.035299		1.132121		Avg 12
164 Di-n-octyl phthalate					1.859592	1.959861	1.709753	1.773981	1.572522	1.566203	1.925721		1.766805		Avg 9.0
166 7,12-Dimethylbenz(a)an					0.534769	0.530442	0.521166	0.567230	0.535520	0.564634	0.588052		0.548831		Avg 4.5
167 Benzo[b]fluoranthene	1.099381	1.412708	1.366837	1.543229	1.299689	1.334471	1.331917	1.473672	1.428103	1.552086	1.546376		1.398952		Avg 9.6
168 Benzo[k]fluoranthene	1.136225	1.679044	1.358458	1.366108	1.222826	1.193511	1.226305	1.263811	1.192898	1.168840	1.289283		1.281574		Avg 12
171 Hexachlorophene					0.052700	0.066959	0.084467	0.085427	0.077154	0.055498	0.074621		0.070975		Avg 18
148 6-Methylchrysene			0.566275	0.553258		0.652006	0.644138	0.675454	0.600364	0.633587	0.524025		0.606138		Avg 8.9

(051-004)

Method: \\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	Level 11	b	M1	M2	%RSD/R^2
169 Benzo[a]pyrene	1.254411	1.374133	1.200307	1.273754	1.120729	1.126316	1.108499	1.201543	1.163919	1.197435	1.195506		1.201505		Avg 6.5
170 3-Methylcholanthrene					0.493025	0.500282	0.527424	0.572074	0.571721	0.583483	0.545098		0.541872		Avg 6.7
173 Dibenz(a,h)anthracene	1.002168	1.230125	1.091409	1.024627	0.893417	0.949816	1.050665	1.113641	1.110867	1.150069	1.062268		1.061734		Avg 8.8
174 Indeno[1,2,3-cd]pyrene	1.521958	1.558104	1.385941	1.259661	1.083215	1.173185	1.288896	1.342756	1.341632	1.388481	1.273468		1.328845		Avg 10
175 Benzo[g,h,i]perylene		Disabled	1.173564	1.063464	0.878929	0.999420	1.074886	1.137780	1.104353	1.161154	1.057218		1.072307		Avg 8.5
177 Dibenz[a,j]acridine					0.634082	0.684207	0.721386	0.710819	0.740672	0.702439	0.709454		0.700437		Avg 4.8
165 Dibenz[a,h]acridine			0.534224	0.606009		0.764851	0.767642	0.897975	0.901418	0.948625	0.916395	-0.57526	0.915952		Linr 0.997
119 Sulfotepp				0.108095	0.107156	0.107909	0.108459	0.111404	0.110662	0.105914	0.111595		0.108899		Avg 1.9

ICalib Error Legend

R1, Curve Coefs Fail the Rule Error Limit Test

RB, Calibration Low Point Fails %Drift Test

(SOP-150)

Attachment 4.

Example: Acceptable Calibration Verification - Form 7 (052-001 to 052-004)

Report Date: 01-Dec-2012 13:12:14 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago
CCVIS, Cal Verification ISTD Check Report

Data File:	\\chi-svr07\chromdata\CMS01\20121128-14073.b\1C1128A.D		
Lims ID:	ccvis	Client ID:	
Inject. Date:	28-Nov-2012 17:22:30	Dil. Factor:	1.0000
Sample Type:	CCVIS		
Sample ID:	CCVIS		
Misc. Info.:	500-0014073-002		
Operator:	DA	Instrument ID:	CMS01
Injection Vol:	1.0 ul	ALS Bottle#:	2
Lims Batch ID:	14073	Lims Sample ID:	2
Sublist:	chrom-1-8270*sub16		
Detector:	MS SCAN		
Method:	\\chi-svr07\chromdata\CMS01\20121128-14073.b\1-8270.m		
Method Label:	TestAmerica Chicago GC/MS SVOA		
Last Update:	01-Dec-2012 13:11:42	Calib Date:	28-Nov-2012 15:17:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\chi-svr07\chromdata\CMS01\20121128-14067.b\STD80.D		
Limit Group:	MSBNA_8270D_ICAL		
Integrator:	RTE	ID Type:	Deconvolution ID
Column Type:	ZB5MS	Column Dia:	0.25 mm
Process Host:	XAWRK018		
Start Cal Date:	30-Dec-2010 12:11:30		
End Cal Date:	28-Nov-2012 15:17:30		

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
$ 7 2-Fluorophenol	1.333099	0.000		1.360455	0.010	2.1	20.0
$ 8 Phenol-d5	1.719380	0.000		1.770149	0.010	3.0	20.0
$ 9 Nitrobenzene-d5	0.433872	0.000		0.435700	0.010	0.4	20.0
$ 10 2-Fluorobiphenyl	1.361583	0.000		1.433754	0.010	5.3	20.0
$ 11 2,4,6-Tribromophenol	0.195553	0.000		0.232028	0.010	18.7	20.0
$ 12 Terphenyl-d14	0.772538	0.000		0.732737	0.010	-5.2	20.0
14 Pyridine	40.0	0.000	41.5	1.543663	0.010	3.6	20.0
13 N-Nitrosodimethylamin	40.0	0.000	42.9	0.731240	0.010	7.2	20.0
16 2-Picoline	1.588857	0.000		1.642947	0.010	3.4	20.0
17 N-Nitrosomethylethyla	0.602174	0.000		0.643199	0.010	6.8	20.0
19 Methyl methanesulfona	0.872569	0.000		0.838552	0.010	-3.9	20.0
21 N-Nitrosodiethylamine	0.622124	0.000		0.622247	0.010	0.0	20.0
22 Ethyl methanesulfonat	1.210468	0.000		1.166900	0.010	-3.6	20.0
24 Phenol	1.932080	0.000		2.042993	0.800	5.7	20.0
26 Aniline	2.206580	0.000		2.226221	0.010	0.9	20.0
28 Pentachloroethane	0.630413	0.000		0.648130	0.010	2.8	20.0
27 Bis(2-chloroethyl)eth	1.420207	0.000		1.382063	0.700	-2.7	20.0
29 2-Chlorophenol	1.523025	0.000		1.512455	0.800	-0.7	20.0

(052-001)

Report Date: 01-Dec-2012 13:12:14 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS01\20121128-14073.b\1C1128A.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
30 1,3-Dichlorobenzene	1.618297	0.000		1.676712	0.010	3.6	20.0
31 1,4-Dichlorobenzene	1.626976	0.000		1.690329	0.010	3.9	20.0
32 Benzyl alcohol	0.930691	0.000		1.005621	0.010	8.1	20.0
33 1,2-Dichlorobenzene	1.571934	0.000		1.644132	0.010	4.6	20.0
34 2-Methylphenol	1.128817	0.000		1.191619	0.700	5.6	20.0
35 2,2'-oxybis[1-chlorop	1.855272	0.000		1.834768	0.010	-1.1	20.0
40 N-Nitrosopyrrolidine	0.713003	0.000		0.730399	0.010	2.4	20.0
39 Acetophenone	1.943586	0.000		2.021754	0.010	4.0	20.0
37 3 4 Methylphenol	1.326903	0.000		1.319408	0.600	-0.6	20.0
38 N-Nitrosodi-n-propyla	1.175772	0.000		1.087006	0.500	-7.5	20.0
41 2-Toluidine	2.271024	0.000		2.427792	0.010	6.9	20.0
42 Hexachloroethane	0.775329	0.000		0.759343	0.300	-2.1	20.0
44 Nitrobenzene	0.431983	0.000		0.437700	0.200	1.3	20.0
46 N-Nitrosopiperidine	0.180296	0.000		0.178982	0.010	-0.7	20.0
47 Isophorone	0.751795	0.000		0.767908	0.400	2.1	20.0
48 2-Nitrophenol	0.210095	0.000		0.218234	0.100	3.9	20.0
49 2,4-Dimethylphenol	0.334401	0.000		0.333101	0.200	-0.4	20.0
50 Bis(2-chloroethoxy)me	0.460994	0.000		0.449707	0.300	-2.4	20.0
52 Benzoic acid	0.169436	0.000		0.165803	0.010	-2.1	20.0
54 2,4-Dichlorophenol	0.294046	0.000		0.318631	0.200	8.4	20.0
55 1,2,4-Trichlorobenzen	0.354947	0.000		0.375034	0.010	5.7	20.0
56 Naphthalene	1.042189	0.000		1.056450	0.700	1.4	20.0
58 2,6-Dichlorophenol	0.303517	0.000		0.337883	0.010	11.3	20.0
57 4-Chloroaniline	0.463271	0.000		0.470977	0.010	1.7	20.0
59 Hexachloropropene	0.263365	0.000		0.297666	0.010	13.0	20.0
60 Hexachlorobutadiene	0.211018	0.000		0.235858	0.010	11.8	20.0
64 N-Nitrosodi-n-butylam	0.268731	0.000		0.267858	0.010	-0.3	20.0
66 4-Chloro-3-methylphen	0.314332	0.000		0.337719	0.200	7.4	20.0
76 Isosafrole	0.514482	0.000		0.536929	0.010	4.4	20.0
68 2-Methylnaphthalene	0.704994	0.000		0.703176	0.400	-0.3	20.0
69 Hexachlorocyclopentad	40.0	0.000	39.0	0.279557	0.050	-2.4	20.0
70 1,2,4,5-Tetrachlorobe	0.657246	0.000		0.674774	0.010	2.7	20.0
72 2,4,6-Trichlorophenol	0.419718	0.000		0.439034	0.200	4.6	20.0
74 2,4,5-Trichlorophenol	0.444746	0.000		0.454402	0.200	2.2	20.0
67 Safrole, Total	0.269095	0.000		0.293430	0.010	9.0	20.0
77 2-Chloronaphthalene	1.289032	0.000		1.297465	0.800	0.7	20.0

(052-002)

Report Date: 01-Dec-2012 13:12:14 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS01\20121128-14073.b\1C1128A.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
79 2-Nitroaniline	0.414060	0.000		0.426944	0.010	3.1	20.0
81 1,4-Naphthoquinone	0.500804	0.000		0.523506	0.010	4.5	20.0
83 1,3-Dinitrobenzene	0.222487	0.000		0.228655	0.010	2.8	20.0
82 Dimethyl phthalate	1.441220	0.000		1.466106	0.010	1.7	20.0
84 2,6-Dinitrotoluene	0.331520	0.000		0.349118	0.200	5.3	20.0
85 Acenaphthylene	2.083840	0.000		2.064196	0.900	-0.9	20.0
86 3-Nitroaniline	0.371722	0.000		0.386726	0.010	4.0	20.0
87 Acenaphthene	1.218503	0.000		1.201483	0.900	-1.4	20.0
88 2,4-Dinitrophenol	0.162365	0.000		0.146378	0.010	-9.8	20.0
89 4-Nitrophenol	0.221417	0.000		0.217770	0.010	-1.6	20.0
90 Pentachlorobenzene	0.551000	0.000		0.610280	0.010	10.8	20.0
92 Dibenzofuran	1.723260	0.000		1.791798	0.800	4.0	20.0
91 2,4-Dinitrotoluene	0.450735	0.000		0.470681	0.200	4.4	20.0
94 1-Naphthylamine	1.235134	0.000		1.287850	0.010	4.3	20.0
95 2,3,4,6-Tetrachloroph	0.324308	0.000		0.356622	0.010	10.0	20.0
96 2-Naphthylamine	1.157959	0.000		1.247307	0.010	7.7	20.0
97 Diethyl phthalate	1.591195	0.000		1.576082	0.010	-0.9	20.0
98 4-Chlorophenyl phenyl	0.662518	0.000		0.715943	0.400	8.1	20.0
99 Fluorene	1.452605	0.000		1.460571	0.900	0.5	20.0
100 N-Nitro-o-toluidine	0.225389	0.000		0.248788	0.010	10.4	20.0
101 4-Nitroaniline	0.354688	0.000		0.402322	0.010	13.4	20.0
102 4,6-Dinitro-2-methylp	0.129100	0.000		0.143049	0.010	10.8	20.0
104 Diphenylamine	0.654668	0.000		0.641108	0.010	-2.1	20.0
103 N-Nitrosodiphenylamin	0.558829	0.000		0.547254	0.010	-2.1	20.0
105 1,2-Diphenylhydrazine	1.645042	0.000		1.710554	0.010	4.0	20.0
108 Diallate	0.297879	0.000		0.291400	0.010	-2.2	20.0
107 1,3,5-Trinitrobenzene	0.245920	0.000		0.268885	0.010	9.3	20.0
109 Phenacetin	0.413976	0.000		0.428957	0.010	3.6	20.0
111 4-Bromophenyl phenyl	0.219467	0.000		0.228468	0.100	4.1	20.0
113 Hexachlorobenzene	40.0	0.000	40.5	0.259557	0.100	1.3	20.0
115 4-Aminobiphenyl	0.717316	0.000		0.769551	0.010	7.3	20.0
117 Pentachloronitrobenze	0.115483	0.000		0.127236	0.010	10.2	20.0
116 Pentachlorophenol	0.102824	0.000		0.112502	0.050	9.4	20.0
118 Pronamide	0.366180	0.000		0.390709	0.010	6.7	20.0
121 Dinoseb	0.178163	0.000		0.204051	0.010	14.5	20.0
123 Phenanthrene	1.153173	0.000		1.131456	0.700	-1.9	20.0

(052-003)

Report Date: 01-Dec-2012 13:12:14 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS01\20121128-14073.b\1C1128A.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
124 Anthracene	1.142128	0.000		1.151290	0.700	0.8	20.0
127 Carbazole	0.975739	0.000		1.007081	0.010	3.2	20.0
130 Di-n-butyl phthalate	1.387438	0.000		1.371762	0.010	-1.1	20.0
135 Isodrin	0.173602	0.000		0.165582	0.010	-4.6	20.0
137 Fluoranthene	1.134879	0.000		1.148664	0.600	1.2	20.0
139 Benzidine	0.717546	0.000		0.686589	0.010	-4.3	20.0
142 Pyrene	40.0	0.000	36.9	1.122779	0.600	-7.8	20.0
143 p-Dimethylamino azobe	0.211142	0.000		0.210335	0.010	-0.4	20.0
144 Chlorobenzilate	0.333465	0.000		0.317931	0.010	-4.7	20.0
146 Butyl benzyl phthalat	0.670397	0.000		0.602010	0.010	-10.2	20.0
147 3,3'-Dimethylbenzidin	0.733578	0.000		0.726751	0.010	-0.9	20.0
150 Kepone	40.0	0.000	39.1	0.073494	0.010	-2.2	20.0
153 2-Acetylaminofluorene	0.501137	0.000		0.476738	0.010	-4.9	20.0
157 3,3'-Dichlorobenzidin	0.425428	0.000		0.427923	0.010	0.6	20.0
156 Bis(2-ethylhexyl) pht	0.975392	0.000		0.885905	0.010	-9.2	20.0
161 Benzo[a]anthracene	1.254187	0.000		1.090840	0.800	-13.0	20.0
162 Chrysene	1.132121	0.000		1.021757	0.700	-9.7	20.0
164 Di-n-octyl phthalate	1.766805	0.000		1.680077	0.010	-4.9	20.0
166 7,12-Dimethylbenz(a)a	0.548831	0.000		0.561725	0.010	2.3	20.0
167 Benzo[b]fluoranthene	1.398952	0.000		1.518263	0.700	8.5	20.0
168 Benzo[k]fluoranthene	1.281574	0.000		1.108583	0.700	-13.5	20.0
169 Benzo[a]pyrene	1.201505	0.000		1.107120	0.700	-7.9	20.0
170 3-Methylcholanthrene	0.541872	0.000		0.532268	0.010	-1.8	20.0
173 Dibenz(a,h)anthracene	1.061734	0.000		1.010716	0.400	-4.8	20.0
174 Indeno[1,2,3-cd]pyren	1.328845	0.000		1.212316	0.500	-8.8	20.0
175 Benzo[g,h,i]perylene	1.072307	0.000		0.982985	0.500	-8.3	20.0
S 178 Total Cresols, TCEQ D			121.8				20.0
S 179 Methyl Phenols,Total			121.8				20.0

(052-004)

Attachment 5.

Example: Sample Corrective Action Report / Run Log Report / Instrument Maintenance Log (053-001 to 053-004)

Instrument ID# _____

TestAmerica Chicago
Corrective Action/Qualification Report GC/MS BNA

CHI-22-21-039/C-08/10

Analytical Methods
___SW846 8270C
___SW846 8270D
___40CFR 625

Batch Name:_____
___Other_____

Instrument Preventative Maintenance was performed per SOP_____

Tune Criteria
Description of Situation:_____

Action Taken:_____

Demonstration of Control:_____

Initial Calibration Criteria
Resolution Checks compared to method criteria: _____
(Benzo(b)fluoranthene & Benzo(k)fluoranthene)
Description of Situation:_____

Action Taken:_____

Demonstration of Control_____

Continuing Calibration Criteria
Resolution Checks compared to method criteria: _____
(Benzo(b)fluoranthene & Benzo(k)fluoranthene)
Description of Situation: _____

Action Taken:_____

Demonstration of Control:_____

Internal Standards (CCV)
Description of situation:_____

Action Taken:_____

Demonstration of Control:_____

Qualification of Data
Data Affected (Client/Sample #)_____

Qualification:_____

Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:_____

Client contacted/NCM

Analyst Signature/date_____/_____

Review Signature/date_____/_____

(053-001)

TestAmerica Chicago
Sample Corrective Action Report for Semi-Volatiles by GC/MS

Client: _____ Job Number: _____ Sublist: _____ TICs: 10 20 30 30+ALK____

Deliverable Rank: 1 2 3 4 Method: a. 625; b. 8270C; c. 8270D Custom Limits: Y / N NCM: _____ Internal Std. Reagent ID_____

Sample ID →											MB	LCS	LCSD
File: _____													
	Matrix: _____ Instrument #: _____ Worklist: _____ Extraction Batch: _____ Analytical Batch: _____												
Dilution													
Int. Std.													
Surrogate													
Spike													
OK													
Files Imported													
ICV AD Batch													
Sample ID →													
File: _____													
	Matrix: _____ Instrument #: _____ Worklist: _____ Extraction Batch: _____ Analytical Batch: _____												
Dilution													
Int. Std.													
Surrogate													
Spike													
OK													
Files Imported													
ICV AD Batch													

(0530a)

Reviewed by: _____ **Date:** _____

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: HSL ICAL-112812 Worklist Num: 14067
Instrument: CMS01 Method: 1-8270
Batch Directory: \\chi-svr07\chromdata\CMS01\20121128-14067.b
Anaylysis Type: SemiVOA Creator: Akcakal, Duran
Inj Volume: 1.00 Inj Vol Units: ul
Run Reagents:
SMISTDWORK_00183, Amount Added: 10.00 , Units: uL

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
# 1 dftpp	500-0014067-001	DFTPP		28-Nov-2012 09:31:30	1D1128.D	1	1.0		sv
# 2 STD05	500-0014067-002	IC	2	28-Nov-2012 11:35:30	STD05.D	2	1.0		sv
# 3 STD01	500-0014067-003	IC	1	28-Nov-2012 11:57:30	STD01.D	3	1.0		sv
# 4 STD1	500-0014067-004	IC	3	28-Nov-2012 12:20:30	STD1.D	4	1.0		sv
# 5 STD2	500-0014067-005	IC	4	28-Nov-2012 12:42:30	STD2.D	5	1.0		sv
# 6 STD5	500-0014067-006	IC	5	28-Nov-2012 13:05:30	STD5.D	6	1.0		sv
# 7 STD10	500-0014067-007	IC	6	28-Nov-2012 13:27:30	STD10.D	7	1.0		sv
# 8 STD20	500-0014067-008	IC	7	28-Nov-2012 13:49:30	STD20.D	8	1.0		sv
# 9 STD40	500-0014067-009	ICIS	8	28-Nov-2012 14:10:30	STD40.D	9	1.0		sv
#10 STD50	500-0014067-010	IC	9	28-Nov-2012 14:33:30	STD50.D	10	1.0		sv
#11 STD70	500-0014067-011	IC	10	28-Nov-2012 14:55:30	STD70.D	11	1.0		sv
#12 STD80	500-0014067-012	IC	11	28-Nov-2012 15:17:30	STD80.D	12	1.0		sv
#13 ICV mix2	500-0014067-013	ICV		28-Nov-2012 15:40:30	ICVMIX2.D	13	1.0		sv
#14 ICV mix1	500-0014067-014	ICV		28-Nov-2012 16:02:30	ICVMIX1.D	14	1.0		sv

(053-003)

TestAmerica Chicago
GC/MS BNA Maintenance Logbook
Instrument No. 23

Page No._____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-Up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Reviewer Signature or Initials / Date:_____ CHI-22-21-040/A-08/10

(053-004)

Attachment 6.

Example: Data Review Checklist
(054-001 to 054-003)

TestAmerica
THE LEADERS IN ENVIRONMENTAL TESTING

Chicago
CHI-22-20-088/D-09/12

GC/MS Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
TUNE:		
Tune passes, all points within the 12 hour clock?		
Reports:		
p-Flag reports reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: CCC's ≤ 30% RSD		
Linear or Quadratic Regression curve fit for all >15% RSD; 20% RSD -8270D; R^2 >0.990		
Plots for all Linear Regressions reviewed?		
conc-intercept < RL (<1/2 RL DoD QSM); < 30% Error (Chrom: Details of Calibration per Analyte)		
Isomeric Pairs reviewed and accepted? (Chrom: View/Documents/Methods/Isomer)		
Responses: SPCCs all pass minimum response factors?		
All manual integrations reviewed were found to be necessary, were performed correctly and have documented reasons?		
ICAL selection of points – documented & performed correctly		
Method:		
Method locked in Chrom?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct and locked in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 70-130% (30%D) criteria? AFCEE-75-125% (25%D)		
QAPP Specific ICV 80-120% (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date _____

Secondary Reviewer: _____ Date _____

(054-001)

TestAmerica Chicago
GCMS Semi-Volatiles (BNA) DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Method: MS BNA: 8270C _____ 8270D_____ 8270 SIM _____ 625 _____

Matrix: WATER / SOIL / SPLP-TCLP / Waste / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Inst# Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: Calib. ID: _____ _____ _____ _____ _____ _____
Sample Hold Time: _____ NCM Ref. Number: _____			
JOB STATUS: 1) Primary Reviewer/Analyst: 2nd Level			
2) Secondary Reviewer/Supervisor: Lab Complete			
View Data Summary: Are all flags correctly assigned? Y N Sample Data Sheets present? Y N Surrogate page present? Y N QC Data Summaries all present? Y N Are samples linked to prep batch? Y N Are all dilutions appearing on summary? Y N NA			
Form 1: IF **original** and **re-run** are to be reported in LIMS: Appropriate suffixes present Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Smp # Original Dilution Comments
FORM 2: Surrogate Recoveries Within Limits Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____
Method Blank Detection Limits Met (<1/2 RL limit for AFCEE / DoD QSM) NCM Ref Number: _____			

(054-002)

TASK	PRI REV	SEC REV	COMMENTS
FORM 5: Tuning Criteria Met/Matches LIMS Analysis Batches ICAL Form 5 OK? Yes No Tunes: _____ _____ _____ _____ _____ _____ NCM Ref Number: _____			Tune time met? Yes No
FORM 8: Internal Standards Criteria Met MPIS or CCVIS NCM Ref Number: _____			List sample numbers:
RAW DATA Verified / Complete			
Form 6: Initial Calibration Criteria met and Complete?			
Form 7: CCV criteria met method criteria NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Tune Yes No 2) Blank Yes No 3) LCS/LCSD Yes No 4) MS/MSD Yes No NA MRL Check Required : Yes No Control Limit Applied: _____			
Manual Integration reports (before and afters) present, analyst initials present and the reasons for the MI are correctly documented and approved?			
Prep Log page Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required / Properly Documented? Yes No			
Additional Comments:			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : _____ Compound: _____

(054-003)

Attachment 7.

Analysis Scheme Flowchart

(Terms defined in the Section 6)

```
              PFTBA
                |
                |
                |
              DFTPP (Form 5)
              /   \ Every 12 hours
             /     \
            /       \
     Initial Calibration--------Calibration Verification
      (Form 6)                  (Form 7)
                    Every 12 hours
                          |
                          |
                         \|/
      [Extraction]------------------Analysis
                          |
                          |
                         \|/
              Operator Data Production/Reduction
                          |
                          |
                         \|/
              Operator Data Packaging/Review
                          |
                          |
                          |
                         \|/
              Final Review/Data Submission
                          |
                          |
                         \|/
                  Report Generation
```

Sample Tracking Flowchart (for EACH unique batch #)

```
                    Sample        \            \                \
                    Receipt--------Log-In--------Cooler---------Prep Group
                    /  |      |   /            /        |       /
                       |      |                         |
          ____________|      |                         |
         |                 Computer            Extraction Batch
         |                    |                         |
         |                    |                         |
        COC                Backlog                      /
 (Chain of Custody)           |                     /Extracts &
         |                    |                   /  Computer
         | \                  |                 /    Hardcopy
        \|/  \              \|/   Refrigerator /          .
         .\    \ __________/_______________/            .
         .  \            /                               .
         .    \        /                                 .
         .      \     /                                  .
         .        \ /                                    .
         .   Base/Neutral Acids                          .
         .         |                                     .
        \./       \|/                                    .
       Filed     Posted     Posted /......................
         \         |         /     \
           \       |       /
             \     |     /
               \   |   /
                 \ | /
                   |
                  \|/
                 Input
        ____________________
        | Sample Tracking Board |
        ____________________
                   |
                   |                  ____
                   |                  | O |  Extraction Record
                   |                  | U |  COC
                  \|/                 | T |
              ANALYSIS----------------| P |  Sequence log
                                      | U |  Comp. Data Prod.
                                      | T |  Tune, Calibration,
                                      ____   QC
                                 (HC) = Hardcopy
                                     |        |
 Operator Review/ ___________________|        |
 Data Reduction  |                            |_______Extracts (Filed by instrument/date of analysis)
     |           |                            |
    \|/          |                            |
 Data Package    |___________                 |_______Archival/Automated DAT back-ups
 w/  review checklist,      \|/                |
CARs_____________________________________________|
     |                                         |
     |1st 100% Review                          |
     |                                         |
    \|/                                        |
 Submission/Final Review_____________________|_______ review checklist, CAR copies filed
     .                                         \
     ._____________________________________________\____Data purged off system
    \./
 Report Generation
```

Attachment 8.

CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations (057-001 to 057-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

Raymond J. Frederici 08/01/2012
Raymond J. Frederici — Date
Quality and EH&S Director

Larry Penfold 08/01/2012
Larry Penfold — Date
Quality Compliance Director

Verl D. Preston 08/01/2012
Verl D. Preston — Date
Quality Assessment Director

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(057-001)

1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

4.5 **Baseline:** The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 **Peak:** An increase in signal from baseline to a maximum and then back to baseline.

4.7 **Coelution:** The concurrent elution of two or more compounds to the detector at the same time.

4.8 **Elution:** The process of movement of compounds from the chromatographic system.

4.9 **Carry-over:** Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 **Peak Tailing:** Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 **Interference Peaks or Fused Peaks:** Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important considerations in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters is necessary to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibrations standards, QC standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. This

process for before & after chromatograms is also required for OVAP-approved laboratories, unless instructed otherwise.

5.4.7.1 Re-integration technique marked, if available, on the data system.

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.

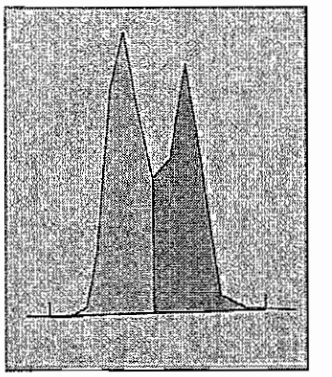
- Expanded section on data miner software to include Chrom Audit Miner
- Included reference to data miner training presentation.

- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples

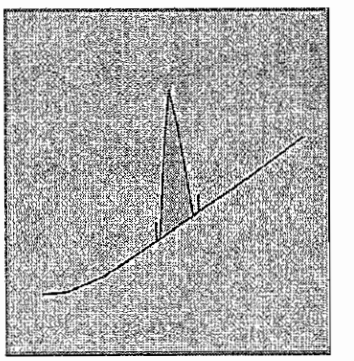
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

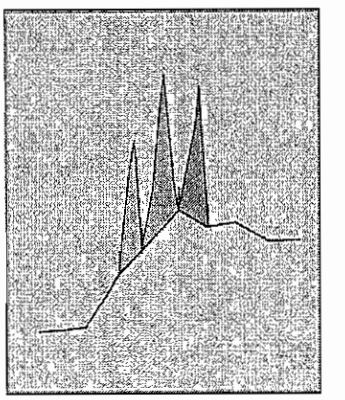
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.

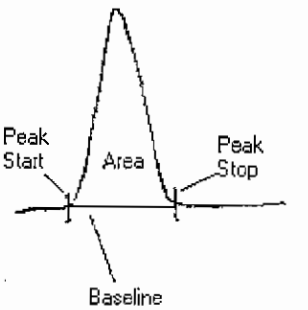
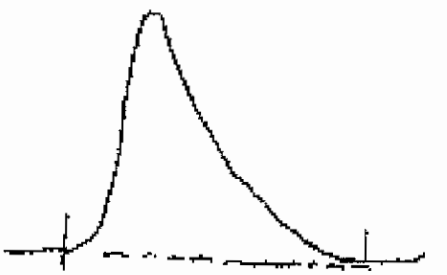


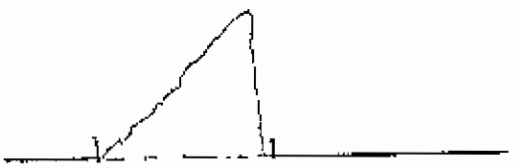
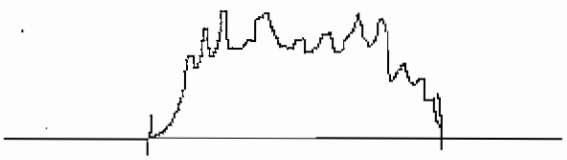
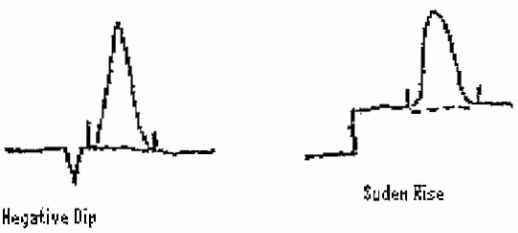
Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.

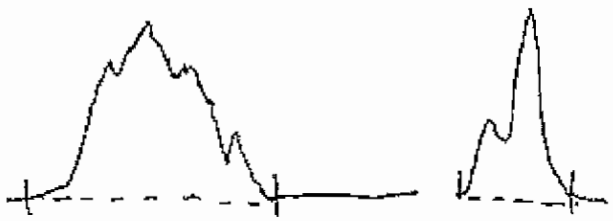
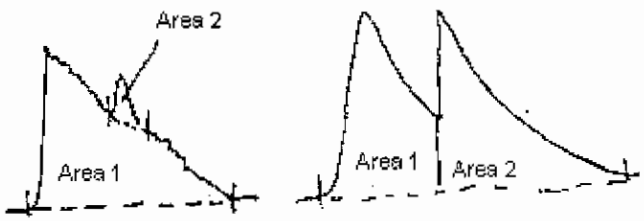


Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.



Ideal peak (Gaussian Shape) The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	

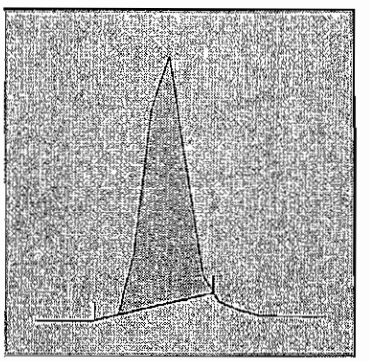
Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

(057-013)

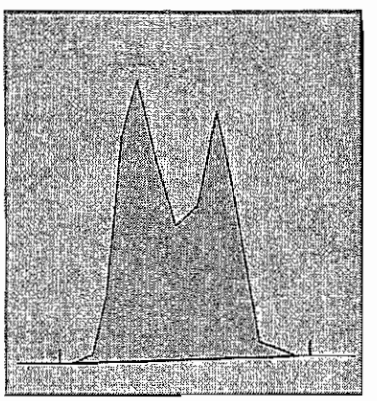
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

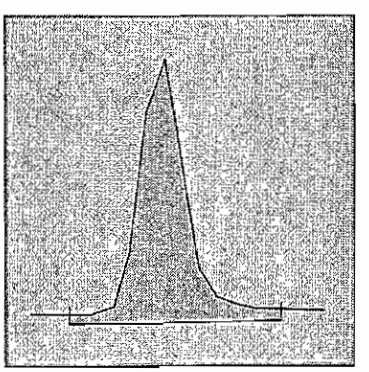
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.

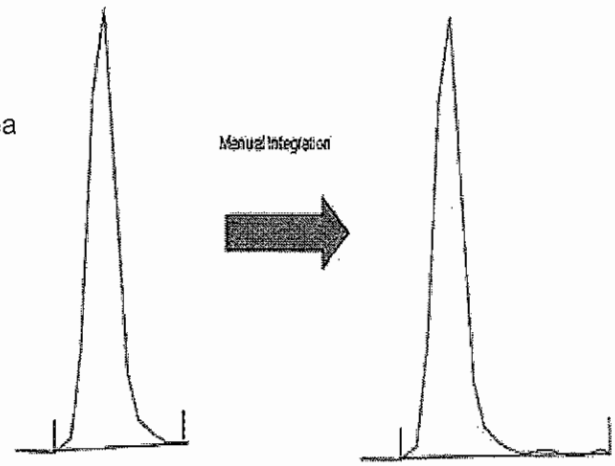


Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.

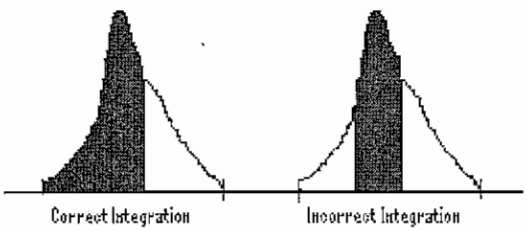


Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.

.



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 9:

Example: ICV recoveries (058-001 to 058-003)

Report Date: 05-Dec-2012 08:41:02 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago
ICV, ICal Verification Report

Data File:	\\chi-svr07\chromdata\CMS01\20121128-14067.b\ICVMIX1.D		
Lims ID:	ICV mix1	Client ID:	
Inject. Date:	28-Nov-2012 16:02:30	Dil. Factor:	1.0000
Sample Type:	ICV		
Sample ID:	ICV mix1		
Misc. Info.:	500-0014067-014		
Operator:	DA	Instrument ID:	CMS01
Injection Vol:	1.0 ul	ALS Bottle#:	14
Lims Batch ID:	14067	Lims Sample ID:	14
Sublist:			
Detector:	MS SCAN		

Method:	\\chi-svr07\chromdata\CMS01\20121128-14067.b\1-8270.m		
Method Label:	TestAmerica Chicago GC/MS SVOA		
Last Update:	28-Nov-2012 16:54:03	Calib Date:	28-Nov-2012 15:17:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\chi-svr07\chromdata\CMS01\20121128-14067.b\STD80.D		
Limit Group:	MSBNA_8270D_ICAL		
Integrator:	RTE	ID Type:	Deconvolution ID
Column Type:	ZB5MS	Column Dia:	0.25 mm
Process Host:	XAWRK005		

First Level Reviewer: rynkarg Date: 28-Nov-2012 16:23:40

Compound	Amount Added	Amount Recovered	%Drift
24 Phenol	50.0	44.9	-10.12
27 Bis(2-chloroethyl)ether	50.0	53.3	6.62
29 2-Chlorophenol	50.0	46.2	-7.65
30 1,3-Dichlorobenzene	50.0	46.7	-6.55
31 1,4-Dichlorobenzene	50.0	47.4	-5.19
32 Benzyl alcohol	50.0	48.4	-3.10
33 1,2-Dichlorobenzene	50.0	47.5	-5.09
34 2-Methylphenol	50.0	46.1	-7.79
35 2,2'-oxybis[1-chloropropane]	50.0	46.7	-6.64
37 3 & 4 Methylphenol	100.0	94.3	-5.67
42 Hexachloroethane	50.0	48.2	-3.65
44 Nitrobenzene	50.0	44.1	-11.81
47 Isophorone	50.0	43.7	-12.67
48 2-Nitrophenol	50.0	45.7	-8.59
49 2,4-Dimethylphenol	50.0	41.9	-16.28
50 Bis(2-chloroethoxy)methane	50.0	44.3	-11.38
52 Benzoic acid	50.0	53.0	6.09
54 2,4-Dichlorophenol	50.0	46.7	-6.56

(058-001)

Report Date: 05-Dec-2012 08:41:02 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS01\20121128-14067.b\ICVMIX1.D

Compound	Amount Added	Amount Recovered	%Drift
55 1,2,4-Trichlorobenzene	50.0	45.3	-9.43
56 Naphthalene	50.0	44.5	-10.91
60 Hexachlorobutadiene	50.0	44.9	-10.29
66 4-Chloro-3-methylphenol	50.0	46.6	-6.75
68 2-Methylnaphthalene	50.0	43.1	-13.73
69 Hexachlorocyclopentadiene	50.0	43.8	-12.34
72 2,4,6-Trichlorophenol	50.0	47.0	-6.06
74 2,4,5-Trichlorophenol	50.0	46.6	-6.88
77 2-Chloronaphthalene	50.0	45.0	-10.02
82 Dimethyl phthalate	50.0	46.3	-7.40
83 1,3-Dinitrobenzene	50.0	48.2	-3.66
84 2,6-Dinitrotoluene	50.0	47.5	-5.02
85 Acenaphthylene	50.0	45.2	-9.56
87 Acenaphthene	50.0	45.1	-9.76
88 2,4-Dinitrophenol	50.0	46.8	-6.33
89 4-Nitrophenol	50.0	45.6	-8.89
91 2,4-Dinitrotoluene	50.0	47.8	-4.37
92 Dibenzofuran	50.0	45.4	-9.16
95 2,3,4,6-Tetrachlorophenol	50.0	50.2	0.47
97 Diethyl phthalate	50.0	46.3	-7.45
98 4-Chlorophenyl phenyl ether	50.0	47.1	-5.73
99 Fluorene	50.0	46.5	-7.00
102 4,6-Dinitro-2-methylphenol	50.0	53.0	6.06
103 N-Nitrosodiphenylamine	50.0	44.6	-10.74
104 Diphenylamine	42.7	38.1	-10.78
105 1,2-Diphenylhydrazine	50.0	47.0	-6.05
111 4-Bromophenyl phenyl ether	50.0	46.1	-7.88
113 Hexachlorobenzene	50.0	44.7	-10.59
116 Pentachlorophenol	50.0	44.7	-10.65
123 Phenanthrene	50.0	44.5	-11.01
124 Anthracene	50.0	44.7	-10.56
127 Carbazole	50.0	47.2	-5.67
130 Di-n-butyl phthalate	50.0	44.8	-10.33
137 Fluoranthene	50.0	46.4	-7.17
142 Pyrene	50.0	41.9	-16.28

(058-002)

Report Date: 05-Dec-2012 08:41:02 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS01\20121128-14067.b\ICVMIX1.D

Compound	Amount Added	Amount Recovered	%Drift
146 Butyl benzyl phthalate	50.0	43.7	-12.54
156 Bis(2-ethylhexyl) phthalate	50.0	43.0	-14.07
161 Benzo[a]anthracene	50.0	40.6	-18.83
162 Chrysene	50.0	40.6	-18.86
164 Di-n-octyl phthalate	50.0	45.9	-8.28
167 Benzo[b]fluoranthene	50.0	45.8	-8.41
168 Benzo[k]fluoranthene	50.0	43.8	-12.41
169 Benzo[a]pyrene	50.0	42.3	-15.31
173 Dibenz(a,h)anthracene	50.0	50.5	0.99
174 Indeno[1,2,3-cd]pyrene	50.0	48.2	-3.66
175 Benzo[g,h,i]perylene	50.0	50.0	-0.03

(058-003)

Appendix A.

DOD QSM Version 4.2: Appendix F QC Requirements Summary Table F-1 and Table F-4 (059-001 to 059-005)

Table F-1 Summary of QC Cheek Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081: Endrin, DDT 8270: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument eonditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of `either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	All positive results must be confirmed.
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance eriteria, the ICAL may not be stable. Results assoeiated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable preeision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average reeovery of the spikes and standard deviation of the replicates must be within designated aeceptanee eriteria. Analysis of field samples may not be conducted until this eheek is suecessful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the preeision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative pereent difference is high, this eould indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistieal proeedures are used to determine the relationship between the signal response and the known eoncentration of analytes of interest. The ICAL must be sueeessful before any samples or other QC check samples can be analyzed.
Internal Standards	A substanee that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable reeoveries in the LCS indieate problems with the preeision and bias of the measurement system. Failure to achieve acceptable reeoveries in a "elean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effeet of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the eorresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correet for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty

(059-001)

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer	Proper tuning of the mass spectrometer must be verified prior to sample analysis

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(059-002)

Table F-4: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12- hour period.	Refer to method speeific ion criteria	Retune instrument and verify. Rerun affected samples	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check DDT (8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for DDT Benzidine & PCP should be present at their normal responses and should not exceed a tailing faetor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<u>1. Average response factor (RF) for SPCCs:</u> VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetraehloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 <u>2. RSD for RFs for CCCs:</u> VOCs and SVOCs ≤ 30% and one option below. **Option 1:** RSD for each analyte ≤ 15% **Option 2:** linear least squares regression: r ≥ 0.995 **Option 3:** non-linear regression-coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
2nd Source calibration verification (ICV)	Once after each ICAL	All project analytes within ± 20% of true value	Correct problem and verify 2nd source standard. Rerun, 2nd source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

(059-003)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
RT window position establishement for each analyte and surrogate	Once per ICAL	Position shall be set using midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Labs may update the RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the lab must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the RTs.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards verification	Every field sample, standard, and QC sample.	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in box D-1; if required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

(059-004)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported, including surrogates	One per prep batch	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For specific analytes(s) in the parent sample, apply J-flag if acceptable criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate MSD or Sample Duplicate	One per prep batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client as to additional action measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for the failed surrogates in the associated prep batch, if sufficient sample material is available. If obvious chromatographic interferences with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interferences.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(C 54-005)

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UP-ME-245.1, Rev. 18 Last Mod ID (circle): NA/ _____

SOP Title: Metals Analysis: Mercury by EPA Methods 245.1/245.5; SW-846 7470A/ 7471A/ 7471B

Affected SOP Section Number(s): 7.7.1 ; 7.3.3
Effective Date of Change: 08/23/13

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO : **Uncontrolled**

Full Signature Approvals Are Kept on File with TestAmerica Chicago Standard Practice Records

Revision Number with Mod ID: UP-ME-245.1, Rv 18B

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change:

   ESI BP LaMP Audit Response.

2. Summary of Procedure Change (circle to indicate if there are attachments t this form: No / Yes: # pages attached = ___)

The following text is to be corrected in Section 7.3.3:

The use of teflon strips for the MB and LCS are required for solid matrix preparations.
The spike solution for the LCS and MS/MSD must be added to the digestion vessel prior to the addition of water.

The following text is to be corrected in Section 7.7.1:

The preparation batch must include the instrument QC: ICV, CCV, CRA, MRL, ICB & CCB.
Note: Do not use the new container ID from the prep batch for the instrument QC in the analytical batch as TALs will not correctly identify or link them correctly to the samples.

David L. Harper 8/22/13
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Lillie Johnson 8/22/13
Approval Signature/Date: Section Manager

Jeanne A. Preston 8/22/13
Approval Signature/Date: QA Manager or Designee

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UP-ME-245.1, Rev. 18 Last Mod ID (circle): NA/ _____

SOP Title: Mercury by Methods....

Affected SOP Section Number(s): 4.0
Effective Date: 11/13/12

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Revision Number with Mod ID: UP-ME-245.1, Rv18A

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1. Reason for SOP Change: TestAmerica corporate audit response

2. Summary of Procedure Change (circle to indicate if there are attachments t this form: No / Yes: # pages attached

To Section 4.0 add the following item :

- 50 mL single-use digestion vessels with caps (check to ensure volume marking at 25 mL is within 2.5% Tolerance)

Diane L. Harper 11/13/12
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Julie Johnson 11/13/12
Approval Signature/Date: Section Manager

Teresa A. Preston 11/13/12
Approval Signature/Date: QA Manager or Designee

CHI-22-09-039/F-11/07

TITLE: Metals Analysis Mercury by EPA Methods 245.1/245.5; SW-846 7470A/ 7471A/ 7471B; and U.S. EPA CLP Doc No. ILM04.0

Approvals (Signature/Date):

Debbie Johnson 10/29/12
Debbie Johnson — Date
Supervisor, Metals

Diane L. Harper 10-29-12
Diane L. Harper — Date
Inorganics Manager

Terese A. Preston 10/29/12
Terese A. Preston — Date
Quality Assurance Manager

Mark Storm 10/29/12
Mark Storm — Date
Env. Health & Safety Coor.

Michael J. Healy 10/29/12
Michael J. Healy — Date
Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analytical procedure for the determination of the mercury concentration in aqueous and non-aqueous media. This SOP was written using EPA 600/4-79-020 Methods 245.1 and 245.5; SW-846, 3rd Edition, Methods 7470A/7471A, and Update IV, 7471B; and U.S. EPA CLP Document No. ILM04.0 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DoD QSM Version 4.2 are located in Attachment 3. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed quarterly for each element by the metals laboratory for each instrument as specified in CLP. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. For DoD QSM, the IDL must be ≤ the Limit of Detection (LOD), which = ½ RL. Refer to IDL SOP, *UP-QA-010* for additional details.

1.1.4 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement.

Matrix	Reporting Limit [1,3]	CRDL [2]
Water	0.2 ug/L	0.2 ug/L
Soil	0.017 mg/kg	0.1 mg/kg

[1] Reporting Limit is used for EPA Method 245.1 and SW-846 7470A/7471A. Reporting Limits may vary depending on sample volume/size, dilution factors, and changes in the MDL.
[2] CRDL (Contract Required Detection Limit) is used for U.S. EPA CLP ILM04.0.
[3] The DoD QSM, version 4.2 and NELAC use the term Limit of Quantitation (LOQ) for the RL.

1.1.5 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

This flameless cold vapor AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and swept from solution and passed through a cell of a double beam AA. Absorbance is a function of mercury concentration.

2.0 INTERFERENCES

- Chloride may be converted to free chlorine during the oxidation step, and since free chlorine absorbs at 253.7 nm, it must be removed before the Hg vapors are swept into the cell. Samples high in chloride, such as brines or certain effluents, may require excess hydroxylamine reagent, up to 6.25 mL, and gentle agitation to prevent chlorine interference. Sulfide and certain volatile organic materials may also interfere with the oxidation step.
- Certain volatile organics may interfere by absorbing at the Hg wavelength, but TestAmerica Chicago does not routinely pre-treat samples for organics.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- To avoid mercury inhalation, the vapors are passed through a charcoal filter and vented.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. 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. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Cmpds.	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

All laboratory glassware used in this test must be acid rinsed, followed by deionized water rinsing.

- 1 Leeman Lab HydraAA Automated Mercury Analyzer, with built in autosampler, running with WinHg Version 1.7 software
- 1 Teledyne Leeman Hydra II_{AA} automated Mercury Analyzer, with built in autosampler, running with Envoy software
- Various printers.
- Class A volumetric glassware
- Eppendorf pipettes

5.0 REAGENTS AND STANDARDS

5.1 Reagents

All reagents and chemicals used in this test must be reagent grade or better.

5.1.1 Miscellaneous Reagents

- Hydrochloric Acid [HCl], Concentrated
- Nitric Acid [HNO_3], Concentrated
- Sulfuric Acid [H_2SO_4], Concentrated
- Deionized (DI) Water, Type II

5.1.2 Sodium Chloride-Hydroxylamine Hydrochloride Solution

Dissolve 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride in sufficient DI water to make 2-liters of solution.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.3 Stannous Chloride Solution

Dissolve 100 g of stannous chloride in 10% hydrochloric acid to make 1-liter of solution.

- Life of Reagent: 1 Month
- Storage Requirements: None

5.1.4 Potassium Permanganate, 5%

Dissolve 175 g of potassium permanganate into 3.5-liters of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.1.5 Potassium Persulfate, 5%

Dissolve 175 g of potassium persulfate into 3,500 mLs of DI water.

- Life of Reagent: 1 Year
- Storage Requirements: None

5.2 Standards

All standards are prepared in Class A volumetric flasks.

5.2.1 Standard Stock Solution I; 1,000 ppm

A 1,000 ppm concentrated mercury standard is purchased from an outside supplier.

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.2 Working Standard Solution I; 100 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, transfer 100 uLs of Stock Solution I to the flask using a 100 uL Eppendorf pipette. Add 2.5 mLs conc. nitric acid as a preservative. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.2.1 Working Standard Solution IA; 25 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, transfer 25 mLs of Working Standard Solution I (Item 5.2.2) to the flask using a graduated cylinder. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking Matrix Spikes, CRAs & the Standard Curve.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.3 Standard Stock Solution II; 1,000 ppm

Purchased from an outside supplier as a 1,000 ppm solution and is from an alternate source than that of Standard Stock Solution I (Rgt. 5.2.1).

- Life of Standard: 1 Year
- Storage Requirements: None

5.2.4 Working Standard Solution II; 200 ppb

To a 1.0 L volumetric flask filled with ~800 mLs DI water, add 2.5 mLs concentrated nitric acid (as a preservative) and 200 uLs of Standard Stock Solution II to the flask (using a 200 uL Eppendorf pipette). Dilute to volume with DI water and invert several times to mix.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.4.1 Working Standard Solution IIA; 50 ppb

To a 100 mL volumetric flask filled with ~80 mLs DI water, add 25 mLs of Working Standard Solution II (Item 5.2.4) to the flask using a graduated cylinder. Dilute to volume with DI Water. Invert and mix to insure complete mixture.

*For use in spiking the ICV/CCV and LCS.

- Life of Standard: 24 Hours
- Storage Requirements: None

5.2.5 Working Standards for Mercury in Water

Standard (ug/L)	mLs of Working Solution IA 5.2.2.1	Final Volume (mLs) Hot Block
Blank	0.0	25
0.2	0.2	25
0.5	0.5	25
1.0	1.0	25
3.0	3.0	25
5.0	5.0	25
CRA (0.2 ug/L)	0.2	25
Matrix Spike (1.0 ug/L)	1.0	25

Standard (ug/L)	mLs of Working Solution IIA 5.2.4.1	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	1.0	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	25
Lab Control Sample (LCS) (2.0 ug/L)	1.0	25

CLP Standard (ug/L)	mLs of Working Solution IIA 5.2.4.1	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	1.0	25
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.5	25

NOTE: ILM04.0 and NELAC require the ICV and CCV to be at different levels.

5.2.6 Working Standards for Mercury in Soil

Standard (ug/L)	mLs of Working Soln. I 5.2.2	Final Volume (mLs) Hot Block
Blank	0.00	50
0.2	0.10	50
0.5	0.25	50
1.0	0.50	50
3.0	1.50	50
5.0	2.50	50
CRA (0.2 ug/L)	0.10	50
Matrix Spike (1.0 ug/L)	0.50	50

Standard (ug/L)	mLs of Working Soln. II 5.2.4	Final Volume (mLs) Hot Block
Init. Cal. Verif. (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50
Lab Control Sample (LCS) (2.0 ug/L)	0.50	50

CLP Standard (ug/L)	mLs of Working Soln. II 5.2.4	Final Volume (mLs) Hot Block
Init. Cal. Verif (ICV) (2.0 ug/L)	0.50	50
Cont. Cal. Verif. (CCV) (1.0 ug/L)	0.25	50

NOTE: ILM04.0 and NELAC require the ICV and CCV to be at different levels.

6.0 CALIBRATION (NON-DAILY)

All calibration procedures are performed on a daily basis. Refer to Section 7.4 for details.

7.0 PROCEDURE

7.1 Quality Control Checks

The following Quality Control samples are performed with each batch of samples. Refer to Section 8.0 for additional details.

QC Sample	Frequency [1]	Control Limits
Method Blank (MB)	1 in 20 samples	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or < LOD (DOD QSM)[3] • < CRDL (CLP)
LCS	1 in 20 samples	• 80-120% Recovery (EPA 245.5 / SW-846 / CLP) • 85-115% Recovery (EPA 245.1)
Matrix Duplicate (DU)[2]	1 in 20 samples	• 20 RPD unless the sample conc. is <5x RL, then ± RL. (EPA 245.1; 245.5 / SW-846) • 20 RPD unless the sample conc. is <5x CRDL, then ± CRDL. (CLP)
Matrix Spike (MS) MS Duplicate (MSD)[2]	1 in 20 samples	• 75 – 125% Recovery unless the sample concentration > spike level by 4x (EPA 245.5 / SW-846 / CLP) • 70 – 130% Recovery (EPA 245.1) • 80 - 120% (DoD QSM) • > 50% Recovery; if <50% Recovery, Method of Standard Additions (MSA) is required (TCLP)

[1] Drinking waters by EPA 245.1; and CLP analyses are analyzed at a frequency of 1 in 10 samples.
[2] The sample selection for MS/MSD or MS/MD, where appropriate, is rotated among client samples so that various matrix problems may be noted and/or addressed. MD's are performed only when requested by the client/project/contract. The MS/MSD are the routinely performed matrix QC indicators.
[3] For DoD QSM v.4.2, ½ the RL = Limit of Detection (LOD)

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and preservations for the referenced programs.

Program	Preservation [1]	Holding Time [2]
SDWA	pH < 2, Cool 4 ± 2°C	28 days VTS [3]
CWA	pH < 2, Cool 4 ± 2°C	28 days VTS
RCRA	pH < 2, Cool 4 ± 2°C	28 days VTS
CLP	pH < 2, Cool 4 ± 2°C	26 days VTSR [4]

[1] Waters are preserved with nitric acid at pH <2; Soils are preserved at Cool 4 ± 2°C.
[2] Holding times include digestion and analysis.
[3] VTS: Verified Time of Sampling.
[4] VTSR: Verified Time of Sample Receipt.

7.3 Sample Preparation

7.3.1 Mercury Water Digestion Procedure - EPA Method 245.1 / CLP ILM04.0

Item	Hot Block
Sample Volume	25 mLs
Reaction Vessel	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	1.25 mLs
Nitric Acid (conc.)	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	2 mLs
Preparation	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	1.5 mLs
Total Volume	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.3.2 Mercury Water Digestion Procedure - SW-846 Method 7470A

Item	Hot Block
Sample Volume	25 mLs
Reaction Vessel	Sample Vials, 50 mLs
Sulfuric Acid (conc.)	1.25 mLs
Nitric Acid (conc.)	0.625 mLs
Potassium Permanganate, 5% Sol. (W/V)	3.75 mLs
Potassium Persulfate, 5% Sol. (W/V)	2 mLs
Preparation	2 hrs. @ 90 - 95°C, Cool
Hydroxylamine Addition	1.5 mLs
Total Volume[1]	34.125 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

[1] A visual check of the digestate final volumes for consistency will be performed. Any volume that is inconsistent with the other sample volumes will be re-digested. This check will be documented in the 'Prep Batch Information' screen in the TALS prep batch.

7.3.3 Mercury Soil Digestion Procedure - SW-846 Method 7471A/7471B

NOTE: A prep batch must be started prior to weighing the solids aliquots. The balance is interfaced with TALS and the sample weights are transferred directly into the ADII batch, eliminating potential transcription errors. The sample IDs can also be scanned directly into the batch from the barcodes on the containers. Three aliquots of soils (~0.2 g each) are combined and digested as one sample, although this requirement has been withdrawn in 7471B. Soil samples are homogenized prior to the removal of the three aliquots according to SOP UP-QA-039, *Quality Assurance – Sample Homogenization and Subsampling Procedures.*

Item	Hot Block
Sample Weight	~ 0.6 – 0.7 grams
Reaction Vessel	Digestion Vessel
DI Water, Type II	2.5 mLs
Aqua Regia, [3:1 HCl (conc.) to HNO_3 conc.)]	2.5 mLs
Preparation	2 min. @ 90-95°C, Cool
DI Water, Type II	25 mLs
Potassium Permanganate, 5% Sol. (W/V)	7.5 mLs
Preparation	30 min. @90-95°C, Cool
Hydroxylamine Addition	3 mLs
Total Volume	50 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.3.4 Mercury Soil Digestion Procedure - EPA Method 245.5 / CLP ILM04.0

NOTE: A prep batch must be started prior to weighing the solids aliquots. The balance is interfaced with TALS and the sample weights are transferred directly into the ADII batch, eliminating potential transcription errors. The sample IDs can also be scanned directly into the batch from the barcodes on the containers.

Item	Hot Block
Sample weight	0.2 - 0.3 grams
Reaction Vessel	Digestion Vessel
Sulfuric Acid (conc.)	5 mLs
Nitric Acid (conc.)	2.5 mLs
Preparation	2 min. @ 90 -95°C, Cool
DI Water, Type II	50 mLs
Potassium Permanganate, 5% Sol. (W/V)	15 mLs
Potassium Persulfate, 5% Sol. (W/V)	8 mLs
Preparation	30 min. @ 90 - 95°, Cool
Hydroxylamine Addition	6 mLs
Total Volume	Dilute to 100 mLs

NOTE: The sample should remain purple for 15 minutes after adding the potassium permanganate. If the sample does not maintain the purple color, a second addition of potassium permanganate is added to all samples of the batch to maintain the purple color.

After the hydroxylamine addition, place the sample digestates on the instrument for analysis, where the reducing agent, stannous chloride, is added by the analyzer from a reservoir to form elemental Hg.

7.4 Calibration / Standardization

Before the instrument is used as a measurement device, the instrument response to known reference materials must be determined, in this case by establishing the working linear curve. All sample measurements must be made within this linear range of the instrument. Periodic known standards and blanks are read throughout the run at the given frequency to verify that the calibration is holding to specified criteria.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. ≥ 0.995
ICV	After the Calibration Curve	• 90 –110% Recovery (EPA 245.5/SW-846/CLP/DoD) • 95 – 105% Recovery (EPA 245.1)
ICB	After the ICV	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or LOD (DOD QSM) • < CRDL (CLP)
CRA	After ICB	• No established limits.
CCV	Every 10 readings; end of each run	• 90 – 110% Recovery (EPA 245.1; 245.5 / CLP) • 80 – 120% Recovery (SW-846, and DoD QSM)
CCB	Every 10 readings; End of each run	• < Reporting Limit (EPA 245.1; 245.5 / SW-846) • < ½ RL, or LOD (DOD QSM) • < CRDL (CLP)

7.5 Preventive Maintenance

The instruments require some routine daily maintenance as well as some scheduled and non-scheduled periodic maintenance. All maintenance will be recorded in the instruments maintenance logbook. The following schedule lists some of the various maintenance procedures and when they should be performed. Additionally, the scheduled maintenance procedures given in the software should be checked and followed.

Equipment	Schedule
Drying Tube	Replace as needed.
Pump Tubing	Weekly, or as needed.
Lamp	Replace as needed (avg. 4 mos. - 1 yr.).
Optical Cell	Clean as needed (typically monthly).
Liquid Gas Separator	Replace every 1-3 yrs., as needed.
Internal Tubing	Should not require replacement under normal circumstances.

7.6 Sample Analysis

7.6.1 Preparing the System

The following procedures must be performed each morning before warming up the system:

- Release the clamps and check the pump tubing for wear.
- Check the reductant volume and rinse volume. Refresh, if needed.
- If the lamp has been off then turn on the lamp power and allow the lamp to warm up for at least 45 minutes for the HydrAA and 20 minutes for the Hydra II_{AA}.
- Start up the system.

7.6.2 Sample analysis

Refer to the work instructions found in Appendices 1 and 2 for details specific to the daily operation of each instrument.

7.7 Documentation

7.7.1 Raw Data and TALS Documentation

The analysis of samples and standards is documented in TALS ADII, supported by the instrument print-out. The digestion batch notes must include the following information, when applicable:

Filter Lot #
HCl Lot #
HNO_3 Lot #
H_2SO_4 Lot #
H_2O_2 Lot #
Potassium Persulfate Lot #
Potassium Permanganate Lot #
Stannous Chloride Lot #
NaCl Lot #
Hood ID
Hot Block ID
Hot Block temperature
Thermometer ID #
Digestion Tube Lot #
Re-pipet Check
Balance ID #

The instrument print-out must be labeled with the instrument identification, analyst signature, date of analysis, and batch ID. All errors and unused data must be clearly identified with appropriate cross-outs, initials, and dates.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. On receipt, each standard is recorded in the TALs reagent module and is issued a unique source ID #. The manufacturer, lot #, date received, expiration date, date of verification, concentration, volume received, and the initials of the recording analyst are entered into the system.

The intermediate standards are prepared daily in according to the instructions in section 5.2. The volumetric is labeled with the standard concentration, the preparatory instructional section of the SOP and states "prepared daily from stock". The date and time of preparation must also be recorded on the flask. The bar-coded stock label is affixed to each volumetric containing an intermediate prepared from that stock. Both stock IDs must be included in the analytical or preparatory batches in the TALS Analysts' Desktop module.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. All data that include real world samples, even entire runs that are not reported, are transferred to TALS. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

The laboratory generates annual statistically generated control limits and these can be used when requested by the client, contract or QAPP. These limits are based on the successive analysis of LCSs.

8.1.1 Calibration curve must be composed of a minimum of a blank and 5-standards. A least square fit linear calibration curve must have a minimum correlation coefficient of 0.995, which must be reported with the raw data.

8.1.2 ICV and ICB will be performed at the beginning of an analytical sequence. The ICV must not vary more than - a) 10% for EPA 245.5, SW-846 including for DoD & CLP methods or b) 5% for EPA 245.1 method from its true value and must be prepared from a different source than the calibration curve standards.

Calibration verification will be performed with a CCV and CCB every 10 samples and at the end of the analysis. The CCV must not vary more than a) 20% for SW-846 methods including DoD or b) 10% for EPA 245.1; 245.5 & CLP methods from its true value and must be prepared from a different source than the calibration curve standards. The CCB must be < Reporting Limit (EPA / SW-846); < ½ RL, or LOD (DOD QSM) and < CRDL (CLP).

8.1.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve (dilute with a digested blank containing all reagents, or repeat the analysis using a smaller sample volume).

8.1.4 A minimum of one MB must be analyzed per sample batch to determine if contamination has occurred

8.1.5 An LCS will be included with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples. The analyzed result must not vary more than 20% from the true value. For EPA Method 245.1, the LCS acceptance limits are 85-115%.

8.1.6 Matrix spike and duplicate samples are analyzed with each batch of 10 (drinking waters and EPA 245.1) or 20 (EPA 245.5, SW-846 or CLP) samples.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, section manager, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, section manager, project manager, QA personnel or the laboratory manager.

Listed below are steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed
- document the problem and the action which was taken to correct the problem in a Non-Conformance Memo (NCM) in TALs LIMs.
- document in the NCM that an in-control situation has been achieved and receive approval of the supervisor, section manager, QA personnel, or the laboratory manager prior to the release of any analytical data associated with the problem.

QC Indicator	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
Initial Calibration Verification (ICV)	• repeat ICV to verify proper preparation; • prepare new ICV from original stock; • recalibrate with a new standard curve; • prepare new stock and/or working standards; • check reagents/solutions and prepare fresh if necessary.
Initial Calibration Blank (ICB)	• prepare new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze ICB and any associated samples.
Laboratory Control Sample (LCS)	If LCS is low: • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set, write a NCM If the LCS is high: • reanalyze LCS to verify that it is out-of-control; • determine the source of error within the preparation procedure, repeat the sample set; • determine if the high result is due to contamination; • check for contamination of reagents, LCS stock solution, or preparation area; • correct for contamination, reanalyze.

QC Indicator	Suggested Corrective Actions
Method Blank (MB)	• reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if the high result is due to contamination; • check for contamination of reagents or preparation area; • correct for contamination, reanalyze set; • in the extreme case where all samples in the set are at least 10X > the MB, reanalysis will not be required. However, a NCM and approval will be necessary.
Matrix Duplicate (DU)	• the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be flagged; • regardless of the outcome of the reanalysis, an NCM will be written and approved by the Supervisor or Section Manager.
Matrix Spike (MS)	• the sample must be reprocessed and reanalyzed; • if the reanalysis results in data that is still out of the control limit, then the sample will be flagged; • regardless of the outcome of the reanalysis, an NCM will be written and approved by the Supervisor or Section Manager.
Continuing Calibration Verification (CCV)	• repeat CCV to verify proper preparation; • prepare new CCV from original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC, especially the CCV, are within the control limits.
Continuing Calibration Blank (CCB)	• prepare new CCB to verify proper preparation; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by the process of elimination, carryover from a previous analysis or reagent contamination and correct the problem, • check reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze CCB and any associated samples; • never dispose of any samples until you are sure that all QC, especially the CCB are within the control limits.
Summary	• If any of the ICV, ICB, CCV or CCB results are out-of-control for any element, the instrument is restandardized and the samples associated with the out-of-control elements are reanalyzed. • If the MB or LCS are out-of-control for any element, the samples are redigested. An exception is if the sample concentrations are ≥ 10X the MB contamination, the results are reported as is. • If any of the DU or MS results are out-of-control, a reanalysis is performed if there is sufficient sample. If there is insufficient sample, or the reanalysis is still out-of-control, the client is notified of the poor results via an NCM. • NCMs are available for out-of-control MB, LCS, MS and DU problems. These are initiated by the analyst performing the analysis. The NCM is then reviewed by the supervisor or section manager. The NCM is stored electronically within the TALS and used to prepare the case narrative (if applicable).

9.0 DATA ANALYSIS AND CALCULATIONS

Perform a linear regression or quadratic fit analysis of the calibration standard results. Compare sample results to the curve to determine the mercury concentration.

9.1 Water ug/L Hg = ug/L x Dilution Factor

9.2 Soil mg/kg Hg = $\frac{(ug/L) \times L \times \text{Dilution Factor}}{wt(g)}$

(Where L = Final digestate volume)

NOTE: All dry weight corrections are made in ADII at the time the batch is calculated.

9.3 Accuracy $\%R = \frac{(A_T - A_O)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_O = Amount recovered in unfortified sample
A_F = Amount added to sample

9.4 Precision $RPD = \frac{|C_1 - C_2|}{(C_1+C_2)/2} \times 100$

Where:
C_1 = First measurement value
C_2 = Second measurement value

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this process goes into the "Corrosive Wastewater" wastestream.
- Single component standards should not be mixed into the waste streams unless approved by the Waste Coordinator. All standards with Hazardous constituents will be turned in to the waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Attachment 1: Example: Instrument Maintenance Log
Attachment 2: Example: Data Review Checklist
Attachment 3: DoD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1 and Table F-7)

Appendix 1: Daily Operation of the HydraAA (HG5)
Appendix 2: Daily Operation of the Hydra II_{AA} (HG6)

14.0 REVISION HISTORY

- Revision 18 updated on 10/29/12
- Annual Review
- Removed drying tube chemical reference

Attachment 1:

Example: Instrument Maintenance Logs (020-001 to 020-002)

TestAmerica Chicago
Hydra AA - Leeman Mercury Analyzer – HG5
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Clean and Refill Rinse Tank							
Clean Sample Tip							
Check/Clean Optical Cell (Clean when reference intensity is <300,000)							

Weekly Maintenance:							
Change Pump Tubing							
Change Activated Carbon and Quartz Wool							

Comments:_____

_____

_____

Return to Control:

_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections

(020-001)

Reviewer Signature:_____ Date:_____

TestAmerica Chicago
Hydra AA II - Leeman Mercury Analyzer – HG6
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Check / Clean and Refill Rinse Bottle							
Check / Clean Sample Tip							
Check/Clean Optical Cell (Clean when reference intensity is <300,000)							

Weekly Maintenance:							
Change Pump Tubing							
Check scheduled maintenance in computer software							

Comments:_____

_____

_____

Return to Control:

_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Comments sections

Reviewer Signature:_____ Date:_____

CHI-22-14-085/A-09/10

(020-002)

Attachment 2.

Example: Data Review Checklist
(021-001 to 021-002)

TestAmerica Chicago
Mercury Data Review Checklist: Automated Cold Vapor

Instrument ID: **HG5** **HG6**

Analytical Batch: _____

Preparation Batch: _____

_____

Copies: _____ _____

_____ _____

_____ _____

_____ _____

****

Method (circle): a. **EPA 245.1** b. **EPA 7470A** c. **EPA 7471A** d. **CLP ILM04.0**

Matrix (circle): a. Drinking Water b. Non-Potable Water c. Soil/Sediment/Waste d. Other_____

Circle: a. Total b. Solubles **QC Type (circle):** a. Standard b. DoD QSM / AFCEE c. CLP d. Other _____

I. Calibration:

NCM #	Analyst	Reviewer	
			1. Calibration is clearly documented.
			a. c.c.: 0.995 to 1.000
			b. y-intercept: **Std. QC:** < RL; **AFCEE/DoD:** < ½ RL; **CLP QC:** < CRDL
			2. Calibration Verification **CLP QC and Drinking waters:** Every 10 Sample Readings **Std. QC and TCLP QC:** Every 20 Sample Readings
			a. ICV/CCV **SW-846:** ± 10% (ICV); ± 20% (CCV); (Note: CCV Default of +/-10% in TALS **CLP:** ± 10% (ICV); ± 10% (CCV) **EPA 245.1:** ± 5% (ICV); ± 10% (CCV)
			b. ICB/CCB **Std. QC**: < RL; **CLP QC**: < CRDL **AFCEE/DoD**: <1/2 RL
			3. CRA **CLP QC**: At CRDL; Analyzed each Calibration; No Limit; **Std. QC**: At CRDL; Analyzed Daily; ± RL

II. Sample Analysis:

NCM #	Analyst	Reviewer	
			Each Preparation Batch:
			a. Must be clearly identified
			b. Contains a maximum of 20 samples
			c. 1 Prep Blank: **CLP:** < CRDL; **Std. QC:** < RL **AFCEE/DoD**: <1/2 RL
			d. 1 LCS: **SW 846/CLP:** 80-120% Rec. **EPA 245.1:** 85-115% Rec.
			e. 1 Matrix Spike: **Std. / CLP:** 75-125% Rec.; ***Unless*** the sample conc. >4X the spike conc. **TCLP:** > 50% Rec.; If <50%, MSA analysis is required **245.1:** 70-130% Rec.
			f. 1 Matrix Spike Duplicate: **Std. / SW 846:** 75-125% Rec.; ***Unless*** the sample conc. >4X the spike conc.; RPD/RSD limits are 20%
			g. 1 Matrix Duplicate: **Std.QC:** RPD/RSD limits are 20% ***Unless*** the sample conc. is <5x RL then ± RL **CLP:** RPD or RSD limits are 20%; ***Unless*** the sample conc. <5x CRDL then ± CRDL applies.

TestAmerica Chicago
Mercury Data Review Checklist: Automated Cold Vapor

I. Data Documentation

Analyst	Reviewer	
		1. The instrument ID must be clearly documented.
		2. If any curve data, or initial or continuing calibration data are outside of the control limits, **an NCM must be written** and the Manager or Supervisor must be notified that re-digestion is required.
		3. Matrix Spike/Matrix Spike Duplicate outside the control limits:
		a. **CLP QC**: Single spike only. No corrective action required, the sample is flagged appropriately.
		b. **Std. QC**: **An NCM must be written** and the Manager or Supervisor must make the decision as to whether re-digestion is required if no matrix spike duplicate was done.
		c. If MSA is performed; check the calculation.
		4. Sample Duplicate outside the control limits:
		a. **CLP QC**: Normally no corrective action required; the result is flagged appropriately.
		b. **Std. QC**: **An NCM must be written** and the Manager or Supervisor must make the decision as to whether redigestion is required.
		5. All unused data is clearly identified.
		6. Standard Traceability is correctly documented.
		7. Data Report accurately reflects the documentation in the Databook and LIMS.
		8. The analyst's full signature is required on the following:
		a. Instrument Raw Data Report
		b. Data Review Checklist
		9. Jobs needing copies are clearly marked
		10. Proper Corrective Action Documentation for any out of control situation is clearly identified.

IV. TALS

Analyst	Reviewer	
		1. Samples Tab:
		a. LIMs Sample IDs/Containers are correct.
		b. Method and Matrix are correct.
		c. Date and Time match raw data.
		d. Dilutions are correct.
		e. Correct suffix designated (where applicable).
		2. Worksheet Tab is complete and correct.
		3. Reagent Tab is complete and correct.
		4. QC Links Tab is correct.
		5. Sample Results Tab:
		a. All unused data are designated Rejected or Acceptable.
		b. All reported analytes are designated Primary or Secondary.
		6. Batch Information Screen: Documentation is complete.
1st	2nd	7. Status set appropriate to review level.

Comments:_____

Analyst Signature: _____ Date: _____.

Reviewer Signature:_____ Date: _____.

Attachment 3.

DoD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1 and Table F-7) (022-001 to 022-005)

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Inorganics (Metals)

QC Check	Definition	Purpose	Evaluation
Calibration Blank	Reagent water containing no analytes of interest.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above the LOD require corrective action
Continuing calibration verification (CCV)	This verificatiou of thc ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for thc analytes are outside the acceptance eriteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this eheck is successful.
Dilution test (Metals only)	Analysis of a positive sample, which has been diluted to a coneentration 1/5th of the original, to confirm that there is no interference in the original sample analysis. (Modified COE)	To assess matrix interference	Agreement within 10% between the concentration for the undiluted sample and 5x the concentration for thc diluted sample indicates the absencc of interferences, and such samples may be analyzed without using the MSA. Results outside acceptance limits indicate a possible matrix effect. For ICP a post-digestion spike must be run.
Duplicate Sample (replicate)	Two identical portions of material collected for chemieal analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzcd identically. (modified QSM)	To provide information on thc heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. Thc ICAL must be successful before any samples or other QC check samples can be analyzed.
Instrument Detection Limit (IDL) Study (6010 and 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide evaluation of instrument sensitivity	IDLs must be established before samples can be analyzed.
Interference check solutions (ICP and ICP/MS only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of thc analytical sequence	No samples can be run if this check does not pass acceptance criteria.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clcan) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in thc LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.

(10 32-001)

F-1 (cont.)			
QC Check	**Definition**	**Purpose**	**Evaluation**
Linear dynamic range or high-level check standards (ICP and ICP.MS only)	High-level check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high=level standard.	The QC check establishes the upper linear range of the calibration.
Low-Level calibration check standard (ICP only)	A reference standard that contains a quantity of analyte equal to or less than the reporting limit.	To confirm the accuracy of measurements at or near the RL.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix Spike (MS)	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
Matrix Spike Duplicate (MSD)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
MSA (ICP only)	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is also called spiking the sample.)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results
Post digestion spike addition (ICP and ICP/MS only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects	To confirm the presence of a matrix interference. Assess matrix effects based on: 1. the occurrence of new and unusual matrices included within the batch, or 2. contingency analysis based on SD or MS failures	To verify the absence of an interference, the spike recovery must be between 75%-125% Results outside the acceptance limits require MSA for all samples within the batch
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(022-002)

DoD QSM Version 4.2: Appendix F - Quality Control Requirements Summary

Table F-7: Inorganic Analysis by ICP and CVAA - Methods 6010 and 7000 Series

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be $\leq$ LOD	NA	NA	Samples may not be analyzed without a valid IDL
Linear dynamic range (LRS) or high-level check standard (ICP only)	Every 6 months	Within $\pm$ 10% of expected value	NA	NA	
Initial Calibration (ICAL) for all analytes ICP: min 1 high std and a Calib.Blank CVAA: min 5 stds and a Calib. Blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used $r \geq 0.995$.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
2nd Source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of 2nd source for all analyte(s) within $\pm$ 10% of true value.	Correct problem and verify 2nd source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing Calibration verification (CCV)	After every 10 field samples and at the end of analysis sequence.	ICP: within $\pm$ 10% of true value. CVAA: within $\pm$ 20% of true value.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard (ICP only)	Daily, after one-point ICAL.	Within $\pm$ 20% of true value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

(022-003)

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank results must not otherwise affect sample results. For common lab contaminants, no analytes detected >RL (see Box D-1)	Correct problem, then see criteria in box D-1; If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration Blank (ICB / CCB)	Before beginning a sample run, after every 10 samples, and at the end of the analysis sequence	No analytes detected > LOD.	Correct problem. Reprep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for the specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are verified trace impurity from one of the spiked analytes) ICS-AB: ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per prep batch	QC acceptance criteria specified by DoD, if available; see Box D-3 and Appendix G.	Correct problem., then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. If the matrix spike fails outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per prep batch per matrix (see Box D-7)	MSD: For matrix evaluation use QC acceptance criteria specified by DoD for LCS. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution Test (ICP only)	One per prep batch .	Five-fold dilution must agree within ± 10% of the original measurement.	ICP: Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50x LOQ.
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery within 75-125% (see Table B-1)	Run all associated samples in the prep batch by MSA or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.

(024-004)

F-7 (cont.)					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method of Standard Addition (MSA)	When matrix interference is confirmed.	NA	NA	NA	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(022-005)

Appendix 1.

Daily Operation of the HydraAA (HG5)
(023-001)

TestAmerica Chicago

HG5: Hydra AA Run Notes

To Start Run

Start - WinHg
Protocol - select water or soil
File - New Dataset - Today's Date - Enter
Sample - Today's Date - Enter

Main Tab
    Reset page #
Control Tab
    Lamp On * needs to warm up for at least 45 min. then
    Gas On
    Pump On
Standard Tab
    New Cal Reset - OK
    DB - Cal Curve - Reset Calib, New Cal, Update Coeffs, Spike Coeffs (Click then hit OK)
    Standard Tab - Click all 6 STD's and Rep 1
Sample Tab
    Click on Rack Icon at Top of page and add samples to 1st column
    Save as today's date
    Sample Tab - Select Rack and Start and Finish
Utility Tab
    DAQ - Do Action - Sample Measurement should be over 300,000
Standard Tab
    Click Stnd Auto to start calibration
Cal Curve Tab on DB
    Click Accept when calibration is finished
Sample Tab
    Click Run Auto to start sample run
Report Tab
    Watch run

When Run is Finished

File - Page Eject - Will not print automatically
Report Tab on DB
    Click on Run (make sure all samples in batch are checked)
    Click on Generate Report
    Format -check PRN File
    Output File Path - click on ... - Double click data folder - type in file name - Generate
    Print Intensities
        Report Spec - Edit - Absorbance - OK
        Generate Report - Generate Report
        Edit - Turn Off Absorbance

Send to LIMS

Data Folder - Find File - right click - send to HG5

Turn Off Instrument

Control Tab
    Lamp - Off
    Gas - Off
    Pump - Off
File – Exit

CHI-22-14-086/A-10/10

(023-001)

Appendix 2.

Daily Operation of the Hydra II_{AA} (HG6) (024-001)

TestAmerica Chicago
HG6: Hydra II Run Notes

To Start Run

Start - Envoy
Method
    Instrument Control
        Lamp - On (Needs to warm up for 20 min. before starting run)
    Calibration
        Clear Calibration
    Sequence
        Sequence – New – Enter/Scan in QC & Samples
        UPDATE
Start Up (Green oval with arrow in it), starts the pump and gas
    Allow instrument to sip stannous and rinse for a few minutes
Run Sequence (Test tubes in a Rack icon)
Analysis
    Right click on first sample
    Insert Chapter – enter date
Method
    Calibration (watch curve)
Analysis
    Results (watch samples)

When Run is Finished

Analysis
    Report
        GENERATE LIMS INPORT
        Statistics – load – Tals import – OK
        Check the chapter folder – Report – CSV File – Type date – Save
        Envoy folder on main screen – Find file – Right Click – Send to TALS
IMPORT
        PRINT REPORT
        Detailed – load – Print – Report – Printer – Type Date – Print

Turn Off Instrument

Click Sleep (Blue oval with black circle inside)
Method
    Instrument Control
        Lamp – Off
Method – Exit

CHI-22-14-087/A-1010

(024-001)

TITLE: Metals Analysis Trace Inductively Coupled Argon Plasma by SW-846 6010B (Simultaneous Operation)

Approvals (Signature/Date):

Debbie Johnson 9/30/13 Debbie Johnson — Date Supervisor, Metals Supervisor	Diane L. Harper 9/30/13 Diane L. Harper — Date Inorganics Manager
Terese A. Preston 9/30/13 Terese A. Preston — Date Quality Assurance Manager	Chris Hoham 9/30/13 Chris Hoham — Date Env. Health & Safety Coor.
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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for determining metal concentrations by Trace Inductively Coupled Argon Plasma (ICAP) Emission Spectrometry - Simultaneous Operation. This SOP was written using U.S. EPA S W-846 "Test Methods for Evaluating Solid Waste", third Edition, Method 6010B as a reference. The hardness calculation found in section 9.4 is from Standard Methods 2340 B-97.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Instrument Detection Limits

Instrument Detection Limits (IDLs) are performed on a quarterly basis for each element and for each instrument. These limits are used to gauge instrument sensitivity and when routinely evaluated, instrument performance without the introduction of method variance can be determined. Refer to the IDL SOP, *UP-QA-010* for additional details.

1.1.4 Reporting Limits

Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values ~3-5x the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the MDL are special circumstances not to be confused with the previous statement. Refer to Table 1 for element wavelength and reporting limits.

1.1.5 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

ICAP is a technique for the analysis of soluble or digested samples for metal concentrations using atomic emission spectrometry. All matrices, including water, TCLP extracts, wastes, soils, sludges and sediments, require digestion prior to analysis. The ICAP 6500 is capable of analyzing simultaneously 29 different elements on a sample, while the Trace 61E analyzes 28 elements simultaneously.

2.0 INTERFERENCES

Spectral, Physical and Chemical Interferences are the three main interferences that are commonly present on the ICAP.

2.1 Spectral Interferences

Spectral interferences are mainly caused by continuous background wavelength, stray light from a high concentration element or overlap of a spectral line from another element. The ICAP can correct for the first two types of interferences by using background correction adjacent to the wavelength. Spectral overlap can be corrected by monitoring the interfering wavelength and computer correcting the results for the false concentration. The values used to correct are known as Inter-Element Correction Factors or IECs. See Section 6.2. Interferent Check Standard A (ICSA) is monitored to detect instrument drift requiring the IEC equations to be corrected.

2.2 Physical Interferences

Physical interferences are usually associated with the sample uptake and nebulization processes. These interferences can usually be eliminated by using a peristaltic pump which assures a constant sample uptake rate. If a sample is extremely viscous or contains a very high dissolved solids concentration, a dilution of the sample may be required to assure a constant and smooth nebulization rate.

2.3 Chemical Interferences

Chemical interferences are normally not significant on the ICAP. These interferences include ionization effects and molecular compound formation. Chemical interferences are highly dependent on the sample matrix type and the element.

Trace ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium may be added to the internal standard solution (100 mLs / 1-Liter).

Most interferences can be corrected by ensuring a constant sample uptake rate and by using the correcting abilities of the computer. If severe interferences are suspected, an alternate method such as ICP/MS may be used to verify the ICAP results.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.
- Parts of the instrument can be extremely hot. Care should be taken if the instrument needs to be adjusted internally.
- Proper ventilation is required due to sample fumes and extreme heat generation (RF generator and plasma) and plasma emissions. People with medical conditions that may respond to ozone emissions should exercise caution.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. 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. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Instrumentation

1) Thermo Jarrell Ash ICAP 61E Trace Analyzer. This simultaneous ICAP currently has 31 analytical wavelengths, with additional wavelengths available. It is equipped with a peristaltic pump for sample uptake and an autosampler, and uses TJA software version 6.2. TestAmerica Chicago Lab ID: ICP5.

2) Thermo Fisher iCAP 6500. Duo, with Cetac ASX-520 autosampler, and Neslab Thermo Flex 900 water chiller. Currently has 32 analytical wavelengths with additional wavelengths available. The instrument employs iTEVA Analyst – TA 2009B (Revision 47) software. TestAmerica Chicago Lab ID: ICP6.

4.2 Supplies

- Volumetric Flasks (Class A): 100 mLs; 200 mLs; 1000 mLs
- Eppendorf Pipettes, varying volumes

5.0 REAGENTS AND STANDARDS

Note: All reagents and blank solutions prepared daily must include the following information on the container labels: date of preparation, method identification, reagent or blank identification, relevant SOP section.

5.1 Reagents

- Milli-Q Water
- *Concentrated Nitric Acid (HNO_3) - InstraPure
- *Concentrated Hydrochloric Acid (HCl) - InstraPure
- *Internal Standards: Indium (In); Ytrium (Y); Profile Standard Arsenic(As) – Inorganic Ventures

*Purchased from a vendor.

5.2 Standards and QC Solutions

All stock standards and QC solutions are purchased from an outside supplier in aqueous form. Two types of standards are used: single element and custom mixed standards. Single element standards are available for most elements at a 1,000 mg/L concentration. The shelf life of all purchased solutions are as stated by the manufacturer and are listed in TALs (LIMS).

5.2.1 Calibration Standards

Prepared with Milli-Q water that has been acidified with 1% HNO_3 and 5% HCl. The calibration standards are prepared daily as follows:

A. Calibration Blank

Add ~500 mLs of Milli-Q water to a 1-L Class A volumetric flask. Repipette 10 mLs conc. HNO_3 and 50 mLs conc. HCl into the flask. Dilute to volume with Milli-Q water and mix thoroughly.

B. Calibration Standards (Refer to Attachment 1 for element concentrations)

Note: A blank and a 3-standard curve is done for each element on the Trace 61E, but a blank and single standard are used to calibrate the iCAP 6500.

The Trace 61E calibration curves must have correlation coefficients of ≥0.995 for 6010B compliance.

Trace 61E Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1A	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S1B	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mLs each of RFW-ICPT-STD-1B RFW-ICPT-STD-1C RFW-ICPT-STD-1D • Dilute to volume with Milli-Q water and mix thoroughly.
S2	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 2.0 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.
S2A	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 0.8 mLs each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3. • Dilute to volume with Milli-Q water and mix thoroughly.
S2B	• Add ~50 mLs of Milli-Q water to a 200 mL Class A volumetric flask. • Re-pipette 2 mLs conc. HNO_3 into the flask. • Re-pipette 10 mLs conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: RFW-ICPT-STD-2A RFW-ICPT-STD-2B RFW-ICPT-STD-3 • Dilute to volume with Milli-Q water and mix thoroughly.

ICAP 6500 Calibration

Standard	Preparation
S1	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 1.0 mL each of: STDIL-STD-1 STDIL-STD-2 • Using an Eppendorf pipette, add 0.50 mL of RFW-ICPT-STD-2B • Using an Eppendorf pipette, add 0.250 mL of RFW-ICPT-STD-2A • Using Eppendorf pipettes, add 0.10 mL each of: Bi, Si and Ag from a 1000 ug/mL stock solution; • Using Eppendorf pipette, add 1.0 mL from Li 100 ug/mL stock solution • Dilute to volume with Milli-Q water and mix thoroughly.
S2	• Add ~50 mLs of Milli-Q water to a 100ml Class A volumetric flask • Re-pipette 1 mL of conc. HNO_3 into the flask. • Re-pipette 5 mLs of conc. HCl into the flask. • Using Eppendorf pipettes, add 2.00 mL of RFW-ICPT-STD-2B • Using Eppendorf pipettes, add 1.00 mL of RFW-ICPT-STD-2A • Dilute to volume with Milli-Q water and mix thoroughly.

5.2.2 Instrument QC Solutions (Refer to Attachment 2 for element concentrations.)

All QC solutions are recorded in the TALS reagent module and are prepared as follows:

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
Reporting Limit Check Std. (ICVL/CCVL)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 1.0 mL of TACHI-1 • 1.0 mL of TACHI-2 • 50 uLs Bi from a 1000 ug/mL stock • 100 uLs Li from 100 ug/mL stock solution • Dilute to volume with Milli-Q water and mix thoroughly.
Initial Calibration Verification (ICV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 8 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 1.6 mLs of 10,000 ug/mL Fe • 1.6 mLs of 10,000 ug/mL Na • 1.68 mLs of 10,000 ug/mL Mg • 3.6 mLs of 10,000 Al • 1.84 mLs of 10,000 ug/mL Ca • 3.6 mLs of 10,000 ug/mL K • Dilute to volume with Milli-Q water and mix thoroughly.

QC Solution	Preparation To a 1-L Class A volumetric flask filled w/ ~500 mLs of Milli-Q water, add the following for each QC Solution:
Continuing Calibration Verification (CCV)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 10 mLs each of: CCV Solution A CCV Solution A1 CCV Solution B • 4.5 mLs of 10,000 ug/mL Al • 2.3 mLs of 10,000 ug/mL Ca • 2.0 mLs of 10,000 ug/mL Fe • 2.0 mLs of 10,000 ug/mL Na • 4.5 mLs of 10,000 ug/mL K • 2.1 mLs of 10,000 ug/mL Mg • Dilute to volume with Milli-Q water and mix thoroughly.
CRI [Contract Required Detection Limit (CRDL) Standard for ICAP]	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 2.0 mL of TACHI-1 • 2.0 mL of TACHI-2 • 100 uLs of 1,000 ug/mL Bi • 200 uLs of 100 ug/mL Li • Dilute to volume with Mill-Q water and mix thoroughly.
ICSA (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A. • Dilute to volume with Milli-Q water and mix thoroughly.
ICSAB (Interferent Check Standard)	• 10 mLs of concentrated HNO_3. • 50 mLs of concentrated HCl. • 100 mLs of CLPP-ICS-A • 10 mLs of CLPP-ICS-B4 • Dilute to volume with Milli-Q water and mix thoroughly.

Note: Some of the volume amounts of the single-element spikes in the above table appear to be reduced from the obvious volume necessary to achieve the final desired concentration. This is to compensate for the presence of those metals in the mixed standards also added to the working standard.

6.0 CALIBRATION (NON-DAILY)

6.1 Linear Range Standard (LRS)

ICAP analysis involves a Linear Dynamic Range (LDR) and a Linear Calibration Range (LCR). At TestAmerica Chicago, the high standard of the LCR is set below the established upper limit of the LDR for all elements. In order to establish, verify, and document linearity at or near that upper limit, a Linear Range Study (LRS) is done every six months on each instrument. This is done by running a single standard at the upper limit of the anticipated linear range of measurement after the instrument has been calibrated in the usual daily manner. The acceptance limits for these single-element verification standards is 95-105%. During routine analysis, all results, target or non-target, that are above the LDR are flagged by the instrument software, aiding the analyst in making appropriate dilution decisions. All samples for which a target analyte or an interfering element result is found to be above the LDR are diluted and re-analyzed until the concentration falls within the instrument's linear range.

6.2 Inter-Element Correction (IEC)

Correction factors for spectral interference will be determined at least annually for all wavelengths used for each analyte reported, any time the ICAP is adjusted in any way that may affect the IECs, or as needed based on continuing observation of Interferent Check Standard A (ICSA) and Interferent Check Standard B (ICSAB). The correction factor is manually calculated from the result ratios between the affected analyte and a known interferent analyzed simultaneously at an appropriately high concentration. The factor, documented with the instrument records, is entered into the method at the instrument PC and becomes part of the software algorithm. See Section 9.5 for calculation details.

Correction factors for spectral interferences other than Al, Ca, Fe, and Mg are recommended and are performed as needed.

7.0 PROCEDURE

7.1 Batch Quality Control Checks

The following section summarizes the quality control (QC) samples associated with ICAP analysis.

QC Sample	Frequency[6]	Control Limit [1]
Method Blank (MB)	1 per 20 samples	≤Reporting Limit (<1/2 RL per QAPP)
Lab Control Sample (LCS) [2]	1 per 20 samples	80 – 120%
Matrix Spike (MS) [3,6]	1 per 20 samples	75 – 125%
MS Duplicate (MSD) [3,5]	1 per 20 samples	75 – 125%; 20 RPD
Duplicates (DU) [4,6]	1 per 20 samples	20 RPD
Serial Dilution (SD)[7] (5x dilution)	1 per 20 samples	± 10% of the original result if analyte conc. >10X MDL[9]
Post Digestion Spike (PDS)[7]	1 per 20 samples – recommended if SD or MS or MSD fails	75 - 125%

[1] Refer to Section 8 for additional details.
[2] LCS Duplicate (LCSD) is run only when required by the client or project.
[3] If sample concentration is ≤4X spike level, 75-125%; if sample concentration is >4X spike level, no control range. If TCLP matrix spike is <50%, Standard Addition must be performed.
[4] If ≥5X reporting limit, 20 RPD; if <5X reporting limit, ± reporting limit; if <reporting limit, no control range.
[5] The sample selection for matrix QC, if not specified by the client or on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed...pre-determined by the digestion department.
[6] Some programs, including IDEM, require 1/10 frequency
[7] The SD and PDS are recommended, but not required per 6010B. TestAmerica Chicago routinely runs the SD, but only runs a PDS upon client request.

7.2 Sample Preservation and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Holding Time[1]	Preservation	Reference
Waters	180 days	HNO_3, pH < 2; Cool 4 ± 2°C	40 CFR Part 136.3
Soils	180 days	Cool 4 ± 2°C	N/A

[1] Inclusive of digestion and analysis.

7.3 Sample Preparation

The most commonly used digestion procedures are SW-846 Methods 3010A (waters) and 3050B (soils). Refer to UP-SP-3000 for details on sample digestion. The samples are received in the metals laboratory as 25, 50 or 100 mL final volumes.

7.4 Calibration / Standardization

7.4.1 Instrument Set Up

Trace 61E (ICP5):

Set up the instrument with the proper operating conditions as defined in the instrument manual. The instrument must be allowed to become thermally stable (~1-hour) prior to profiling and calibration. The instrument is profiled using a 1-ppm Arsenic standard by aspiration and selecting the automatic profile feature from the Thermo Fisher software. The peak position reading should be within ± 0.1. If the reading is acceptable, record the peak area in the maintenance logbook & rinse. If the reading is > ± 0.1, set the micrometer to the adjusted vernier position given by the instrument and profile again to verify. Record the peak area in the maintenance logbook and rinse. The instrument is now ready to calibrate.

ICAP 6500 (ICP6):

Set up the instrument with the proper operating conditions as defined in the instrument manual. The instrument must be allowed to become thermally stable (~1/2-hour) prior to calibration. Alignment of the torch or sensor chip is done as needed. A 2ppm Zinc solution is aspirated to align the torch after cleaning. An autopeak adjust is run only if some part of the induction system is changed such as pump speed, nebulizer pressure, etc.

7.4.2 Standardization

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the linear range of the instrument.

The Trace 61E instrument is standardized using a calibration blank and 3 calibration standards, the 6500 uses a calibration blank and one standard. The results are given in intensities.

Standard	Frequency	Control Limit
Calibration Curve	Initially	Corr. Coeff. ≥ 0.995
High Standards (S1, S2)	After the Calibration Curve	± 5% of the Known Conc.
Initial Cal. Verif. (ICV)	After the Calibration Curve	± 10% of the Known Conc.
Initial. Cal. Blank (ICB)	After the ICV	≤Reporting Limit (≤1/2 RL per QAPP)
CRI	Daily	None Required
MRL	Daily in DoD batches	80-120%
ICSA / ICSAB	Daily	*± 20% of the Known Conc. or absolute value <RL for all non-spiked elements. (<1/2 RL per QAPP)
Cont. Cal. Verif. (CCV)	Every 10 reading; End of each run	± 10% of the Known Conc.
Cont. Cal. Blank (CCB)	Every 10 readings; End of each run	≤Reporting Limit(≤ ½ RL per QAPP)

* Note: Samples with extremely high "A" concentrations and low "B" concentrations may warrant further evaluation.

7.5 Preventive Maintenance

The required preventive maintenance is listed in the preventive maintenance logbooks which are kept at the instruments. All maintenance is recorded in these logbooks along with the date and the signature of the analyst performing the maintenance. Documentation of 'Return to Control' must be entered into the maintenance logbook. The instruments are under a full service contract with the manufacturer for all major repairs and an annual PM service. A copy of the service maintenance record must be attached to the maintenance logbook.

7.5.1 Daily Maintenance

Includes changing the pump tubing for consistent sample uptake, a visible check of the waste container to make sure that it doesn't overflow, and checking the tip of the torch for excessive buildup of material.

7.5.2 Monthly Maintenance

Includes cleaning and checking the water re-circulator for proper fluid level, cleaning the spray chamber and nebulizer if needed. Check the air filters to be cleaned or replaced.

7.6 Sample Analysis

7.6.1 Analytical Run

After the instrument is standardized (Section 7.4.2), an analytical run is initiated. The first run of the day would proceed as follows:

- S1,S2 Reanalysis of calibration standard as a sample
- ICV Initial Calibration Verification
- ICB Initial Calibration Blank
- CRI Spiked Blank Sample
- ICSA Interferent Check Standard A
- ICSAB Interferent Check Standard B
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank
- MRL Spiked Blank Sample (DoD)
- MB (1) Method Blank
- LCS (2) Laboratory Control Sample
- Sample (3)
- Sample (4) Serial Dilution (SD)
- Sample (5) Matrix Duplicate (DU)
- Sample (6) Matrix Spike (MS)
- Sample (7) Matrix Spike Duplicate (MSD)
- Sample (8)
- Sample (9)
- Sample (10)
- CCV Continuing Calibration Verification
- CCB Continuing Calibration Blank

If the CCV and CCB results are acceptable, the run may continue without restandardization. If any of the post-run QC is out of control, or close to being out of control, the instrument is restandardized before analyzing the next batch. Any samples with elements associated with an out of control CCV or CCB will be reanalyzed.

7.7 Documentation

7.7.1 Instrument Sequence-Log

The analysis of samples and standards is documented within the instrument sequence log (Attachment 3) in the TALs LIMs system and is supported by the instrument print-out.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into LIMS and is issued a unique source ID#. The manufacturer, lot #, date received, expiration date, date of verification and the initials of the recording analyst are also entered. The preparation data for all intermediate standards prepared in house are also entered into TALS. Unique bar-coded labels are generated that are affixed to the stock or standard containers. The unique standard IDs are scanned or manually entered into the TALS preparation and analytical batches along with the volumes used for spiking the various QC standards. The standard IDs appear in the TALS-generated raw data.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 4) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors
- Results outside of calibration range

8.0 QUALITY CONTROL

8.1 QC Summary

NOTE: The following laboratory acceptance criteria are set at default control limits. Statistical limits are generated on an annual basis from cumulative LCS data and can be implemented when specified by the client, contract, or QAP.

8.1.1 Method Blank (MB)

At least one MB and one LCS will be included in each digestion batch of 20 samples. The MBs are analyzed to determine if contaminants are being introduced into the sample via the sample preparation procedures.

8.1.2 Laboratory Control Sample (LCS)

The LCS is analyzed to determine the accuracy of the digestion process.

Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be within ±20% of the known concentration. If the LCS results are outside these control limits, all samples in the preparation set must be redigested and reanalyzed. Refer to Attachment E for element concentrations.

8.1.3 Matrix Duplicate (DU)

A duplicate sample will be prepared at a frequency of 5% (1 in 20 samples). A 20 RPD is set as the acceptance limits.

8.1.4 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

The MS / MSD will be prepared at a frequency of 5% (1 in 20 samples). The recovery must be within 75–125%. (Exception allowed if the sample concentration exceeds 4 times the spike added concentration.)

TCLP - If the MS recovery is <50% and the concentration does not exceed the regulatory limit or the sample concentration is within 20% of the regulation level, the Method of Standard Addition (MSA) is required. Three aliquots of the sample are spiked at 50%, 100% and 150% of the sample concentration or, if the sample concentration is < RL, the MSA is at 50%, 100% and 150% of the MS level. The data is subjected to linear regression whereas the concentration of the unknown is the x-intercept and the correlation coefficient value must be $\geq$ 0.995.

8.1.5 Serial Dilution

A Serial Dilution (5X) will be prepared from the digestate at a frequency of 5% (1 in 20 samples). If the concentration is >10 times the MDL, results should agree within +/- 10% of the original results.

8.1.6 Post Digestion Spikes

A Post Digestion Spike may be analyzed to compensate for the matrix effect. Post Digestion Spikes are analyzed on a project, program or client request basis. Recoveries must fall between 75-125%.

8.2 Corrective Action

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out- of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The analysis must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, or QA personnel.

The following steps that must be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out-of-control situation were addressed;
- document the problem and the action which was taken to correct the problem on a NCM;
- document on the NCM that an in-control has been achieved; and
- receive approval (signature) of the supervisor or QA personnel prior to the release of any analytical data associated with the problem.

QC Indicator	Suggested Corrective Actions
Calibration Curve	• reanalyze the standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICV	• repeat the ICV to verify proper preparation; • prepare a new ICV from original stock; • recalibrate with a new standard curve; • prepare a new stock and/or working standards; • check the reagents/solutions and prepare fresh if necessary.
ICB	• prepare a new ICB to verify proper preparation; • verify that the instrument base-line is stable and perform necessary maintenance, cleaning, etc.. to achieve stability; • determine the source of contamination by process of elimination, carryover from a previous analysis or reagent contamination and correct the problem; • check the reagents/solutions and prepare fresh if necessary; • correct for any contamination and reanalyze the ICB and any associated samples.
LCS	If the LCS is low: • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • If continued out of control, redigest and reanalyze the set. • Write an NCM. If the LCS is high: • reanalyze the LCS and all samples in the set for the failed analyte(s) to confirm that it is out of control. • check for contamination of reagents, LCS stock solution, or in the preparation area; • correct for contamination, redigest and re-analyze the set; • Write an NCM.
MB	• reanalyze the MB to verify that it is beyond the reporting limit; • determine the source of contamination; • determine if a high value is due to contamination; • check for contamination of reagents or in the preparation area; • correct for contamination, reanalyze the set; • report results at least 10x > the MB or < RL where applicable; • in the extreme case where all samples in the set are at least 10x > the MB or < RL for all metals, reanalysis will not be required; however, an NCM will be written and approved by the supervisor or section manager.
DU	• an NCM will be written and approved by the supervisor or section manager.
MS / MSD	• an NCM will be written and approved by the supervisor or section manager.
Serial Dilution (SD)	• prepare a new serial dilution to verify proper preparation; • an NCM will be written and approved by the supervisor or section manager.
CCV	• repeat the CCV to verify proper preparation; • prepare a new CCV from the original stock; • check for instrument base-line drift or a change in one or more of the reagents; • check the reagents/solutions and prepare fresh if necessary; • recalibrate with a new standard curve and repeat all samples since the previous in control CCV; • never dispose of any samples until you are sure that all QC are within the control limits.

QC Indicator	Suggested Corrective Actions
CCB	• check reagents/solutions to verify proper preparation and prepare fresh if necessary; • verify that the instrument base-line is stable and/or perform necessary maintenance, cleaning, etc., to achieve stability; • correct for any contamination (carryover from a previous analysis or reagent contamination) and reanalyze the CCB and any associated samples; • never dispose of any samples until you are sure that all QC are within the control limits.
Additional CAs	• If any of the ICV, ICB, ICSA, ICSAB, CCV or CCB results are out-of-control for any element, the instrument is re-standardized and the samples associated with the out-of-control elements are reanalyzed, unless certain exception criteria are met. • If the MB or LCS is out of control for any element, the samples are redigested. An exception is if the sample concentrations are ≥ 10X the MB contamination or < RL. In this case, the results are reported as is. • If any of the MD or MS/MSD results are out of control, the client is notified of the poor results via a case narrative that is sent with the data report. • NCMs can be created at any time in LIMS, most frequently as the data are initially reviewed in AD. They are automatically forwarded to the appropriate managers as emails and the text appears in the final report to the client.

9.0 DATA ANALYSIS AND CALCULATIONS

The sample results are stored in a data file on the desktop computer. The data is transferred over to LIMs and edited there. This system helps to eliminate transcription errors, since data is not entered by hand.

9.1 Accuracy

9.1.1 ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

9.1.2 MS / MSD % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

9.2 Precision (RPD)

9.2.1 Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

9.3 Concentration

mg/kg or L = $\frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in the TALs LIMs analytical batch at the time the data is processed.

9.4 Calculations for SiO2 and Hardness

Calculation of SiO2

(Si) Silicon Raw (mg/L) * 2.1392 * PF

PF=Prep Factor

Calculation of Magnesium Hardness

(Mg) Magnesium Raw (mg/L) * 4.118 * PF

Calculation of Calcium Hardness

(Ca) Calcium Raw (mg/L) * 2.497 * PF

Calculation of Total Hardness

(((Ca) Calcium Raw (mg/L) * 2.497) + ((Mg) Magnesium Raw (mg/L) * 4.118)) * PF

9.5 Calculation of Inter-element Correction Factors

Spike a blank with the known interfering element at an appropriate concentration, typically at the upper level of the linear range. For ICP5, divide the observed result of the affected element by the observed result of the interfering element. Since the IEC study was done with the previous IEC factor applied, add (or subtract if negative) this result to the current correction factor value in the ICP5 software. The result is the new IEC for the affected element, equal to the adjustment made on 1 ppm of the affected element. The ICP6 software has an auto-IEC function that can be applied optionally.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1.0, 7.0 and 8.0.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Table 1	Element and Reporting Limits
Attachment 1(A&B)	Standard Stock Solutions
Attachment 2	Stock QC Solutions
Attachment 3	Example: Analysis Sequence Log / Maintenance Log
Attachment 4	Example: Data Review Checklist
Attachment 5	Known Digested Quality Control

14.0 REVISION HISTORY

- Revision 16 updated on 09/30/13
- Remove reference to the DoD QSM Criteria
- Updated sections 5.2.1 and 5.2.2 with the Li Standard Preparation instructions
- Section 7.5 was updated to include the include vendor service maintenance records in the logbook.
- Table 1, Attachment 1B; Attachment 2 and Attachment 5 were updated to include Li

Table 1.

Element and Reporting Limits

Element	ICAP 61E (ICP5) Wavelength (nm)	ICAP 6500 (ICP6) Wavelength (nm)	Reporting Limit (RL)[1] (ug/L)	Soils (mg/kg)
Al	308.2	308.2	200	20.0
Sb	206.8	206.8	20	2.0
As	189.0	189.0	10	1.0
Ba	493.4	455.4	10	1.0
Be	313.0	234.8	4	0.40
Bi	N/A	223.0	20	2.0
B	249.6	208.9	50	5.0
Ca	317.9	317.9	200	20.0
Cd	226.5	228.8	2	0.20
Cr	267.7	267.7	10	1.0
Co	228.6	228.6	5	0.50
Cu	324.7	324.7	10	1.0
Fe	271.4	271.4	200	20.0
Pb	220.3	220.3	5	0.5
Li	N/A	670.7	10	1.0
Mg	279.0	279.0	100	10.0
Mn	257.6	257.6	10	1.0
Mo	202.0	202.0	10	1.0
Ni	231.6	231.6	10	1.0
K	766.4	766.4	500	50.0
Se	196.0	196.0	10	1.0
Si	288.1	212.4	200	20.0
Ag	328.0	328.0	5	0.50
Na	588.9	589.5	1,000	100
Sr	421.5	421.5	5	0.50
Tl	190.8	190.8	10	1.0
Sn	189.9	189.9	40	4.0
Ti	334.9	334.9	5	0.50
V	292.4	292.4	5	0.50
Zn	206.2	206.2	20	2.0
Y [3]	371.0	224.3/360.0/371.0	N/A	N/A
In[3]	N/A	230.6	N/A	N/A

[1] These are routine ICAP reporting limits (RL). Lower RLs may be available to use per client request. Sample RLs will vary depending on sample volume, dilution factors, and changes in MDLs. Contact the laboratory for the most current RLs based on annual MDL determinations.
[3] Y, and In on the iCAP 6500, are used as internal standards and are introduced continuously to all samples (including standards and QC samples) via the peristaltic pump at an approximate concentration of 5 ppm. Trace ICP can have some ionization effects caused by torch positioning. To eliminate these effects, Cesium may be added to the internal standard solution (100 mLs / 1-Liter).

Attachment 1A.

Trace 61E Standard Solutions, in mg/L

Vendor	Stock Name	Element	Conc. (mg/L)	S1A	S1B	S1	S2A	S2B	S2
Inorganic Ventures	RFW-ICPT-STD-1B	Sb	100	0.4	0.5	1			
		Mo	100	0.4	0.5	1			
		Si	100	0.4	0.5	1			
		Sn	100	0.4	0.5	1			
		Ti	100	0.4	0.5	1			
Inorganic Ventures	RFW-ICPT-STD-1C	Al	1,000	--	5	10			
		Fe	1,000	--	5	10			
		K	1,000	--	--	--			
		Na	1,000	--	--	--			
		Li	800	3.2	4	8			
		Mg	800	--	4	8			
		Ca	400	--	--	--			
Inorganic Ventures	RFW-ICPT-STD-1D	As	100	0.4	0.5	1			
		Ba	100	0.4	0.5	1			
		Be	100	0.4	0.5	1			
		Bi	100	0.4	0.5	1			
		B	100	0.4	0.5	1			
		Cd	100	0.4	0.5	1			
		Cr	100	0.4	0.5	1			
		Co	100	0.4	0.5	1			
		Cu	100	0.4	0.5	1			
		Pb	100	0.4	0.5	1			
		Ni	100	0.4	0.5	1			
		Se	100	0.4	0.5	1			
		Ag	100	0.4	0.5	1			
		Sr	100	0.4	0.5	1			
		Tl	100	0.4	0.5	1			
		Zn	100	0.4	0.5	1			
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000				40	50	100
		K	10,000				40	50	100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000				20	25	50
		Fe	5,000				20	25	50
		Mg	5,000				20	25	50
		Na	5,000				20	25	50
Inorganic Ventures	RFW-ICPT-STD-3	Pb	2,000				8	10	20
		Mn	1,000				4	5	10
		V	1,000				4	5	10

Note: Not all the above standard concentrations for Al, K, Ca, Fe, Mg, NA, and Pb are used for calibration.

Attachment 1B.

iCAP 6500 Calibration Stock Standard Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	S1 mg/L	S2 mg/L
Inorganic Ventures	STDIL-STD-2	Sb	100	1	
		Mo	100	1	
		Sn	100	1	
		Ti	100	1	
Inorganic Ventures	STDIL-STD-1	As	100	1	
		Ba	100	1	
		Be	100	1	
		B-	100	1	
		Cd	100	1	
		Co	100	1	
		Cr	100	1	
		Cu	100	1	
		Mn	100	1	
		Ni	100	1	
		Pb	100	1	
		Se	100	1	
		Sr	100	1	
		Tl	100	1	
		V-	100	1	
		Zn	100	1	
Inorganic Ventures	CGBI1-1	Bi	1000	1	
	CGSI1-1	Si	1000	1	
	CGAG1-1	Ag	1000	1	
Inorganic Ventures	RFW-ICPT-STD-2A	Al	10,000		100
		K	10,000		100
Inorganic Ventures	RFW-ICPT-STD-2B	Ca	5,000		100
		Fe	5,000		100
		Mg	5,000		100
		Na	5,000		100
Inorganic Ventures	MSLI-100ppm	Li	100	1	

Attachment 2.

Example of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICV (mg/L)	CCV (mg/L)
High Purity	CCV Solution A	As	50	0.4	0.5
		B	50	0.4	0.5
		Ba	50	0.4	0.5
		Be	50	0.4	0.5
		Bi	50	0.4	0.5
		Cd	50	0.4	0.5
		Co	50	0.4	0.5
		Cr	50	0.4	0.5
		Cu	50	0.4	0.5
		Ni	50	0.4	0.5
		Pb	50	0.4	0.5
		Se	50	0.4	0.5
		Fe	500	20	25
		Mn	500	4	5
		V	500	4	5
		Tl	50	0.4	0.5
		Zn	50	0.4	0.5
		Sr	50	0.4	0.5
High Purity	CCV Solution A1	Ca	200	20	25
		Li	400	---	---
		Na	500	20	25
		Al	500	40	50
		Mg	400	20	25
		K	500	40	50
High Purity	CCV Solution B	Ag	50	0.4	0.5
		Sb	50	0.4	0.5
		Mo	50	0.4	0.5
		Si	50	0.4	0.5
		Sn	50	0.4	0.5
		Ti	50	0.4	0.5
Ultra	Single Elements	Al	10,000	40	50
		Ca	10,000	20	25
	* spiked on top of custom mixes.	Fe	10,000	20	25
		Na	10,000	20	25
		K	10,000	40	50
		Mg	10,000	20	25

Attachment 2.
(continued)
Examples of Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	CRI Conc. (mg/L)	MRL conc. (mg/L)
Inorganic Ventures	Aluminum	Al	10,000	0.4	0.2
	Antimony	Sb	1,000	0.04	0.02
	Arsenic	As	1,000	0.02	0.01
	Barium	Ba	1,000	0.02	0.01
	Beryllium	Be	1,000	0.008	0.004
	Bismuth[2]	Bi[1]	1,000	0.04	0.02
	Boron	B	1,000	0.1	0.05
	Cadmium	Cd	1,000	0.004	0.002
	Calcium	Ca	10,000	0.4	0.2
	Chromium	Cr	1,000	0.02	0.01
	Cobalt	Co	1,000	0.01	0.005
	Copper	Cu	1,000	0.02	0.01
	Iron	Fe	10,000	0.1	0.2
	Lead	Pb	1,000	0.01	0.005
	Lithium	Li[2]	100	0.02	0.01
	Magnesium	Mg	10,000	0.2	0.1
	Manganese	Mn	1,000	0.02	0.01
	Molybdenum	Mo	1,000	0.02	0.01
	Nickel	Ni	1,000	0.02	0.01
	Potassium	K	10,000	1.0	0.5
	Selenium	Se	1,000	0.02	0.01
	Silicon	Si	1,000	0.4	0.2
	Silver	Ag	1,000	0.01	0.005
	Sodium	Na	10,000	2.0	1.0
	Strontium	Sr	1,000	0.01	0.005
	Thallium	Tl	1,000	0.02	0.01
	Tin	Sn	1,000	0.08	0.04
	Titanium	Ti	1,000	0.01	0.005
	Vanadium	V	1,000	0.01	0.005
	Zinc	Zn	1,000	0.04	0.02

[1] Bismuth is run on ICP6 only.
[2] Lithium is run on ICP6 only.

Attachment 2.
(continued)
Stock QC Solutions

Vendor	Stock Name	Element	Conc. (mg/L)	ICSA Conc. (mg/L)
Inorganic Ventures	CLP Interferent "A" Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
				ICSAB Conc. (mg/L)
Inorganic Ventures	CLP Interferent "A" Solution	Al	5,000	500
		Ca	5,000	500
		Mg	5,000	500
		Fe	2,000	200
Inorganic Ventures	CLPP-ICS-B4	Cd	100	1
		Ni	100	1
		Zn	100	1
		Sb	60	0.6
		Ba	50	0.5
		Be	50	0.5
		Co	50	0.5
		Cr	50	0.5
		Cu	50	0.5
		Mn	50	0.5
		V	50	0.5
		Ag	20	0.2
		As, Tl	10	0.1
		Pb, Se	5	0.05

Attachment 3.

Example: Analysis Sequence Log / Maintenance Log (025-001)

TALS - TestAmerica Chicago - [Analyst Desktop II - 204697]

File View Window Tools Help

Edit | Print | Find | Doc's | FAQ | Settings | Help

Batch: 204697 - Method: 6010B - Equipment: ICP5

#			Sample Lims Sample ID	Date	Time	Dil	Matrix
1			S0	09/27/2013	15:08	1.0	
2			S1A	09/27/2013	15:14	1.0	
3			S1B	09/27/2013	15:21	1.0	
4			S1 500-204697/4	09/27/2013	15:27	1.0	
5			S2 500-204697/5	09/27/2013	15:34	1.0	
6			S2A	09/27/2013	15:39	1.0	
7			S2B	09/27/2013	15:44	1.0	
8			S1 500-204697/8	09/27/2013	15:51	1.0	
9			S2 500-204697/9	09/27/2013	16:00	1.0	
10			ICV 500-204697/10	09/27/2013	16:08	1.0	
11			ICB 500-204697/11	09/27/2013	16:15	1.0	
12			CRI 500-204697/12	09/27/2013	16:21	1.0	
13			ICSA 500-204697/13	09/27/2013	16:27	1.0	
14			ICSAB 500-204697/14	09/27/2013	16:33	1.0	
15			CCV 500-204697/15	09/27/2013	16:42	1.0	
16			CCB 500-204697/16	09/27/2013	16:48	1.0	
17			MB 500-202737/1-A (500-2376652)	09/27/2013	16:54	1.0	
18			LCS 500-202737/2-A (500-2376653)	09/27/2013	17:00	1.0	
19			500-62997-B-1-A (500-2376654)	09/27/2013	17:07	1.0	Digestat
20			500-62997-B-1-A SD 5 (500-23766	09/27/2013	17:13	5.0	Digestat
21			500-62997-B-1-B DU (500-2376655)	09/27/2013	17:19	1.0	Digestat
22			500-62997-B-1-C MS (500-2376656	09/27/2013	17:25	1.0	Digestat
23			500-62997-B-1-D MSD (500-237665	09/27/2013	17:31	1.0	Digestat
24			500-62997-B-2-A (500-2376658)	09/27/2013	17:38	1.0	Digestat
25			500-62997-B-3-A (500-2376659)	09/27/2013	17:44	1.0	Digestat

Run Log | Sample Quants | Sample List | Worksheet | Reagents | Batch Results | Sample Results | Conditions Review | QC Links | Warnings

Ready | Calculate | Auto-link QC: On | Auto-reject: Off

TestAmerica Chicago | XAWRK022 | Preston1 | CORPXS.TAI.COM:Chicago | Session Time: 0 day(s), 08:50:55

TestAmerica Chicago
TJA Trace ICAP (61E) – ICP5
Instrument Maintenance Log

Page No._____

	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials	Date/Initials
Daily Maintenance:							
Check/Change Pump Tubing							
Check Waste Container							
Check Torch for buildup (Note Cleaning)							
Check Nebulizer / Spray Chamber							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							
Internal Standard Filename / As =							
Internal Standard Filename / Y =							

Non-Routine Maintenance: **	Date	Initials	Maintenance Details	Return to Control

Comments:_____

_____

Any Maintenance/Repair/Part Replacement performed that is not listed above must be documented in the Non-Routine Maintenance sections

Reviewer Signature:_____ Date:_____

CHI-22-14-063/C-09/12

(025-001)

Attachment 4.

Example: Data Review Checklist
(026-001 to 026-002)

TestAmerica Chicago
ICAP Metals Data Review Checklist

Instrument ID: ICP 5 ICP6 ICP8 **Filename:**_____

Analyst Initial(s):_____ **LIMS Batch No.:**_____

Copies: _____

_____

****

Method (circle): a. **EPA 200.7** b. **SW-846 6010B** c. **SW-846 6010C**
Matrix (circle): a. Drinking Water b. Non-potable water c. Soil/Sediment/Waste d. Other_____
Circle: a. Total b. Solubles **QC Type (circle):** a. Standard b. Project QAPP c. Other

I. Calibration:

Analyst	Reviewer		NCM #
		1. Verification of standard traceability and expiration (daily).	
		2. Calibration is clearly documented:	
		a. Instrument is calibrated using ICP5: A Blank and three Calibration Standards; ICP6 & ICP8: A Blank and one Calibration Standard The correlation coefficient must be ≥0.995 (6010B); ≥ 0.998 (6010C) The | y-intercept | ≤ RL; (≤ ½ RL per Project QAPP)	
		b. Reanalysis of the top calibration standard as a sample. Control limits are 95 - 105%. (Run once daily prior to sample analysis).	
		3. Calibration Verification: (10% Frequency):	
		a. ICV/CCV: **6010B/6010C** – 90-110% **EPA 200.7 (ICV)** – 95-105% ICVL/CCVL: **6010C** - 70 – 130%	
		b. ICB/CCB: **Std. QC:** < RL; **SW-846 QC:** < 3x MDL.; **Project QAPP**: <½ RL	
		4. **Std. QC:** Analyzed at the beginning of the day and every 8 hours thereafter:	
		a. CRI: 2x CRDL; No Limit Set	
		b. ICSA/ICSAB: 80-120%	
		5a. MRL: **Project QAPP:** 80-120% or per QAPP **6010C:** 10% frequency, 70-130% 5b. DLCK: **Project QAPP:** 10 – 190%	

II. Sample Analysis:

Analyst	Reviewer		NCM #
		1. Each Prep Batch consists of a maximum of 20 samples of a similar matrix:	
		a. Prep Batches must be clearly identified	
		b. 1 Prep Blank **Std. QC** - < RL **TCLP** - < TCLP RL **Project QAPP** <1/2 RL	
		c. 1 LCS **Std.** - 80-120% Rec.; **EPA 200.7** - 85-115% Rec.	
		d. 1 Duplicate **Std.** - RPD or RSD limits are 20%; ***Unless*** the sample conc. is <5x RL the ±RL applies. EPA 200.7 – 10% Frequency	
		e. 1 Matrix Spike **Std.** 75-125% unless sample conc. exceeds MS conc. by 4x; **200.7** – 70-130% - 10% frequency; **Project QAPP** – 80-120%	
		f. Analytical MS **TCLP** - >50% (MSA performed if <50% recovery)	
		g. Serial Dilution 1 per 20 samples; 10% Difference Limit	
		h. A post-digestion spike (PDS) must be performed for **200.7** (85-115%) if the above limits are not met. Program or QAPP specific criteria may apply.	
		i. Turbidity Checked: **EPA 200.7 Drinking Water** (< 1 NTU; no prep required).	
		j. **EPA 200.7**: Sample dilution performed for analytes > 90% of linear range. Sample dilution is performed for analytes or interferents > Linear Range Check	
		k. Results for samples that are diluted agree with the original analysis.	

TestAmerica Chicago
ICAP Metals Data Review Checklist

III. Data Documentation

Analyst	Reviewer	
		1. Raw Data:
		a. Unused data is clearly identified.
		b. All crossed out data is initialed and dated.
		c. Out of control QC is clearly identified.
		d. Any data that has a tick is commented on with appropriate action taken.
		e. The first page of the run must have the filename; instrument; and analyst's signature

		2. Run Log:
		a. Unused data is clearly identified.
		b. All cross outs are initialed and dated.
		c. Analyst's Signature is required.

IV. LIMS

Analyst	Reviewer	
		1. Samples Tab: a. LIMs Sample IDs/Containers are correct.
		b. Method and Matrix are correct.
		c. Date and Time match raw data.
		d. Dilutions are correct.
		e. Correct suffix designated (where applicable).
		2. Worksheet Tab is complete and correct.
		3. Reagent Tab is complete and correct.
		4. QC Links Tab is correct.
		5. Sample Results Tab: a. All unused data are designated Rejected or Acceptable.
		b. All reported analytes are designated Primary or Secondary.
		6. Batch Information Screen: Documentation is complete.
1st	2nd	7. Status set appropriate to review level.

Comments:

_____

_____

_____

_____

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

CHI-22-14-074/I-08/13 (026-002)

Attachment 5.

Known Digested QC Values (mg/L)

Element	LCS/Spike	TCLP Spike
Al	2	---
Sb	0.5	---
As	0.1	5
Ba	2	100
Be	0.05	---
Bi	0.5	---
B	1	---
Cd	0.05	1
Ca	10	---
Cr	0.2	5
Co	0.5	---
Cu	0.25	0.25
Fe	1	---
Pb	0.10	5
Li	0.5	---
Mg	10	---
Mn	0.5	---
Mo	1	---
Ni	0.5	0.5
K	10	---
Se	0.10	1
Si	5	---
Ag	0.05	1
Na	10	---
Sr	1	---
Tl	0.10	---
Sn	1	---
Ti	1	---
V	0.5	---
Zn	0.5	---

Default Control Limits
LCS: 80 - 120%
Spike: 75 - 125%
TCLP Spike: >50%

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

TP 3/21/13

Original SOP Number/Revision #: UP-MV-8260,Rev.24 3 Last Mod ID (circle): NA/ _____

SOP Title: Gas Chromatography / Mass Spectrometry – Volatiles: SW-846 Method 8260B

Affected SOP Section Number(s): 9.1.1
Effective Date: 03/18/13

CONTROLLED DISTRIBUTION

COPY # :
ISSUED TO :

Full Signature Approvals Are Kept on File with TestAmerica Chicago Standard Practice Records

Revision Number with Mod ID: UP-MV-8260, Rev 24A 23 nr 3-21-13

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change: BP LaMP - ESI Audit Response.

2. Summary of Procedure Change (circle to indicate if there are attachments t this form: No / Yes: # pages attached = ___)

Section 9.1.1 Added text as follows:

SOILs_High Level Methanol:

SW-846 8000C, Section 11.10.5 states the following:

Solid samples with a significant moisture content (>10%), designated for volatile organic analysis, that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. This total volume is then expressed as V_t in the sample concentration calculations provided. Therefore, in order to report results for volatiles analysis of samples containing significant moisture content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as V_t. The total solvent/water volume is calculated as follows:

uL solvent/water V_t = [mL of solvent + ((%moisture/100) x g of sample)] x 1000 uL/mL

Dry Weight Concentration ug/kg = $\frac{(X_s)(V_t)(D)}{(W_s)}$ / (%Solids / 100)

X_s = Calculated concentration of the analyte (ng/uL) in the sample.
V_t = Total volume of the concentrated extract (uL)
D = Dilution Factor
W_s = Weight of the sample extracted or purged (g)

Generally, it is recommended that the calculated concentrations of volatile organics samples that are solvent extracted in a water-miscible solvent/water dilution effect for situations when the sample moisture content is greater than 10%. The potential under reporting of volatile concentrations is more pronounced as the percent moisture content increases.

3-21-13
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

3/21/13
Approval Signature/Date: Section Manager

Jessica A. Preston 3/21/13
Approval Signature/Date: QA Manager or Designee

TITLE: Gas Chromatography Mass Spectrometry - Volatiles SW-846 Method 8260B

Approvals (Signature/Date):

11-30-12 JoAnn Petruszak-Kmetty — Date Supervisor, GC/MS VOA	12/3/12 Mark Storm — Date Env. Health & Safety Coor.
12/3/12 Terese A. Preston — Date Quality Assurance Manager	12/1/12 Michael J. Healy — Date Laboratory Director

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1.0 SCOPE / APPLICATION

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) using SW-846 Methods 8260B and 8000B as references. The preparation of all volatile samples is based on Methods 8000B and 5030B. Method 5035 is covered by a separate SOP (UP-SP-5035), but can also be found in this SOP.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Specific requirements pertaining to the DOD QSM Version 4.2 are located in Attachment 10. These requirements are additionally applicable to all NFESC projects. Any deviations from these procedures and/or variances from must be addressed appropriately in accordance with standard operating protocol and pre-approved on a project by project basis.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC and DOD QSM documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on a quarterly basis to meet the requirements of the DoD QSM version 4.2.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits [a.k.a., Estimated Quantitation Limits (EQLs) as designated in the method] are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported.

Table 1 defines the reporting limits and analyte list for SW-846 Method 8260B.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 2.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials. The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then, temperature ramped to separate the compounds and introduce them to the source. The mass filter separates the ions, which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Instrument calibration occurs about every 12-hours, or prior to analysis. Instrument maintenance is performed as needed or daily basis.

2.0 INTERFERENCES

1. External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation including rinsing of all volumetric glassware with methanol/water, the rinsing of syringes with water, the baking of both for a minimum of 30 minutes, and proper sample handling and instrument maintenance should eliminate these sources. The rinse water bottles that are filled with Milli-Q water will be rinsed with Methanol on a monthly basis or more frequently as needed to prevent biologic growth within the bottles. A laboratory method blank (MB) is analyzed prior to any analysis to show absence of any contaminants. Reagent (Milli-Q) water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

2. Carryover is also another source of contamination. Any time a high-level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. If the carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e., trap replacement), it is documented in the maintenance logbook. Refer to Section 7.4 for information on preventive maintenance.

3. Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

4. The volatile lab must be free of solvents. All analytes must be less than their RLs or < 3X the RL for Acetone and Methlyene Chloride. The volatile lab is under positive pressure to reduce lab contamination; however, intermittent low levels of acetone and methylene chloride may occasionally be detected. Refer to Section 8.2 (Corrective Action) for clarification for blank contamination.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- All employees will adhere to the practices and policies in the TestAmerica Corporate Safety Manual (CSM) and will read the MSDS's for the materials used in this method before handling or using the material.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Current Hardware/Software

- 4 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column.
- 1 Hewlett-Packard 7890A GC interfaced with a 5975C MSD. Equipped with DB-624 column.
- 2 Hewlett-Packard 6890 GC interfaced with a 5975 MSD. Equipped with DB-624 column.
- 2 Tekmar 3000 concentrators, 1 Enchon concentrator, 5 OI Elcipse 4660 concentrator in connection with 8 Varian Archon Autosamplers for 8 systems.
- 1 Encon Evolution concentrator in connection with 1 EST Centurion Autosampler.
- 2 Hewlett-Packard 5890 GC interfaced with a 5972 MSD. Equipped with DB-624 column used as Screeners.
- 8-Hewlett-Packard Chemstations and peripheral hardware.
- 1-Hewlett-Packard Chemserver 9000 series with Target/NT version 4.14
- Chrom version 1.2

The GC/MS has a temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng is analyzed.

4.2 Data System

The analytical systems are interfaced with stand alone PC's which are Pentium based systems running Agilent Chemstation. This system is capable of continuous acquisition and storage of mass spectral data. The software allows plotting specific masses versus time or scan numbers (Extracted Ion Current Profile-EICP) and integration of that abundance. The system also stores the data. The system contains the latest NBS Library.

4.3 Data File Name/ Batch Directory Assignment

Tune, standard, blank, and laboratory control sample (LCS) data files are designated by specific letters unique to each instrument in conjunction with the appropriate month and day (example : 3b0318 = instrument #3, first 12 hour BFB tune, March 18). When a worklist is made in Chrom, a unique Chrom ID# is assigned to each file which aids in the transfer of data from chemstation to Chrom.

4.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL luer-lock gas-tight syringes
- assorted purge vessels (water, 5/25 mL)
- top-loading balance, capable of weighing to ± 0.1 g, stainless steel spatula
- assorted amber and clear Teflon-lined screw-capped vials (1.5-2.0 mL, 3.5-5.0 mL)
- cleaned 40 mL vials w/Teflon-lined screw-caps
- assorted volumetrics (10 mL, 20 mL, 25 mL, 50 mL and 100 mL)

5.0 REAGENTS AND STANDARDS

The majority of the calibration standards are EPA certified, A2LA or second-source verified by the standard vendor in situations where suitable SRMs (Standard Reference Material) was available. For those compounds where standards must be made from neat material (due to instability) or some non-routine compounds, **where available**, a second-source is purchased and used in the LCS to verify the standard

Each time a new initial calibration is required, new standards are prepared and the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds and venders).

All neat standards received are entered into TALs (LIMS). A label is printed from TALs and placed on the bottle. All neat standards are then stored according to manufacturer's recommended storage conditions. The standard is issued a unique ID# [i.e., Neat Standards Reference Number (NSRN)] which is used to track all standards as they are used as is or in preparation of stock/working solutions. The format of the standards in TALs (LIMS) will prevent working or intermediate level solutions from being used past the expiration date of the neat or stock solutions. Lot # of Methanol reagent used should be recorded in the comment section of the prep batch in TALs. In addition, the preparation of the 20% Sodium Bisulfate should be recorded in TALs.

5.1.1 Reagent Water (Milli-Q)

1-Liter of water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are < EQL or 3x EQL for methylene chloride and acetone.

5.1.2 Methanol (MeOH)

All new lot numbers of P & T J.T.Baker Methanol are analyzed and verified to be free of contaminants. This information is available on the TestAmerica Oasis Web-Site that can be accessed by all analysts. The currently approved lot numbers are listed on this site.

5.2 Surrogate Spiking Solution

Working surrogates are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following surrogates are used:

Compound	Concentration
4-Bromofluorobenzene	\
1,2-Dichloroethane-d_4	250 ppm
Toluene-d_8	/
Dibromofluoromethane	

Upon arrival to the laboratory, the standard is entered into TALs (LIMS). When the ampule is opened, the transfer to the surrogate vial on the Archon Autosampler is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier). A label is printed from TALs and placed on the instrument above the surrogate vial.

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 14 days or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of one year unopened. *

5.3 Internal Standard Spiking Solutions

Working internal standards are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following internal standards are used:

Compound	Concentration
Pentafluorobenzene	\
Chlorobenzene-d_5	250 ppm
1,4-Difluorobenzene	/
1,4-Dichlorobenzene-d_4	

After opening, the mixture is transferred to the internal standard vial on the Archon autosampler. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier). A label is printed from TALs and placed on the instrument above the internal standard vial.

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 14 days or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of 1-year unopened. *

Working Internal Standards/Surrogate mix are also prepared using the custom mixes above and diluting to 50 ppm as follows:

Custom Mix	Volume (mL)	MeOH	Concentration
250 ppm Internal Standard Mix	4	Dilute to 20 mLs	50 ppm each component
250 ppm Surrogate Mix	4		

For waters, 5uL injected into 5 mL of water/sample results in 50 ppb internal and surrogate concentrations.

- The mixture is transferred to and stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: Working IS/SS solutions have an expiration date of 2-weeks.

Purgeables
Calibration Standards

VOC Custom Mix (No Gases)
100 ug/mL in MeOH

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2-Trichlorotrifluoroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,1-Dichloropropylene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
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-Chlorotoluene
4-Chlorotoluene
4-Isopropyltoluene
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorobenzene
Chloroform
Chlorohexane
cis-1,2-Dichloroethylene
cis-1,3-Dichloropropylene
Dibromomethane
Dibromochloromethane
Dichloromethane
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
m-Xylene
n-Butylbenzene
n-Propylbenzene
Methyl-tert-butyl ether
Naphthalene
o-Xylene, p-Xylene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethylene
Tetrahydrafuran
Toluene
trans-1,2-Dichloroethylene
trans-1,3-Dichloropropylene
Trichloroethylene

ICAL 2 STD Custom Mix
100 ug/ml in MeOH
2-Methylnaphthalene
1,3,5-Trichlorobenzene
1,3-Butadiene
Isopropylether
Methyl Acetate
Hexane
Heptane
Cyclohexane
Ethyl ether
Methyl Cyclohexane
Chloroprene
2,2,4-Trimethylpentane

APIX Custom STD
2000, 8000 & 10000 ug/mL
Allyl Chloride
Ethyl Methacrylate
Methyl Methacrylate
Methacrylonitrile
Pentachloroethane
Trans-1,4-Dichloro-2-Butene
Iodomethane
Isobutanol
Cyclohexanone
n-Butanol
2-Nitropropane
Ethyl Acetate

Gaese Mix
100 ug/mL in MeOH
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride
Dichlorofluoromethane

Vinyl Acetate
2000 ug/mL in MeOH

Volatile Custom Ketone Mix
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
Carbon Disulfide
100 ug/mL in MeOH

Nitriles and Acrolein Custom Mix
Acetonitrile 800 ug/mL in MeOH
Acrylonitrile 800 ug/mL in MeOH
Propionitrile 800 ug/mL in MeOH
Acrolein 4000 ug/mL in MeOH

2-Chloroethylvinylether
2000 ug/mL in MeOH

5.4 Stock Purgeable Standards

These are obtained as custom mixes from Restek and Supelco. The contents of each solution and concentration appear on the previous page. Upon opening, all contents are transferred to 1.5-2.0 mL amber, Teflon-lined screw-capped vials. Listed are compounds in the EPA TCL and includes compounds done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier. See appropriate entries in TALs (LIMS).

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, 1-year is assigned to un-opened ampules. This is applicable for all "neat" standards.

5.4.1.1 Main 8260 Mix

The 8260 Working Standard is a vial transfer:

Compound / TCL Mix	Volume[1] (mL)	Concentration
8260 Custom Mix Stock/Working Std	2	100 ppm each component

5.4.1.2 Gases

The Gas Working Standard is a vial transfer:

Compound / TCL Mix	Volume (mL)	Concentration
Gases Mix	2	100 ppm each component

5.4.1.3 Additional Compounds

The Ketone and Acrolein/Nitrile Working Standards are vial transfers:

Compound / TCL Mix	Volume[1] (mL)	Concentration
Ketone Custom Mix Stock/Working Std	2	100 ppm each component

Compound / TCL Mix	Volume[1] (mL)	Concentration
Nitriles/Acrolein Custom Mix Stock/Working Std	2	800 ppm Nitriles 4000 ppm Acrolein

The CEVE and Vinyl Acetate Working Standard are prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
2000 ppm CEVE	100	Dilute to 2 mLs	100 ppm each component
2000 ppm Vinyl Acetate	100		

5.4.1.7 Appendix IX

The Appendix IX and extra compounds are prepared as follows:

Compound / TCL Mix	Volume[1] (mL)	MeOH	Concentration
APIX Custom Stock Standard	250ul	Diluted to 2mLs	250/1250/10000 ppm

The ICAL 2 Custom Working Standard is a vial transfer:

Compound / TCL Mix	Volume[1] (mL)	Concentration
ICAL2 Custom Mix Stock/Working Std	2	100 ppm each component

- Life of Standard: Unopened ampules are assigned the manufacturer's expiration date or 1 year from receipt. Working solutions have an expiration date of 1-week (Gases, 2-CEVE, Vinyl Acetate, Acrolein and nitriles) and 2-weeks for all others.
- Storage Requirements: These mixtures are stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark.

NOTE: All standard 'recipes' are listed here in this SOP for guidelines for standard preparation. These 'recipes' are subject to change. The standards listed here are standards that are regularly used. Some clients may request other compounds not listed here. Those compounds will be evaluated for accuracy via this method and analyzed for on a project basis.

5.4.1.8 Low Level Standard

A low level standard is prepared by making a 1/10 dilution of the stock standards of each of the above (nitriles and acrolein included). This standard is used to prepare the low points in the initial calibration. The low level standard may contain the Main 8260 Mix, gases, nitriles and acrolein, and any other required standard. A low-level standard for the Appendix IX compounds is also prepared separately due to duplication of some compounds.

A low level surrogate solutions is also prepared by a 1/10 dilution of the working for low points in the water curve. The calibration levels may vary with the compounds. See recipes in the calibration section for the levels. The low point in the calibrations is based on each compounds reporting limit.

All solutions are stored in a 1.5-2.0 mLs amber Teflon-lined screw-capped vials at –10°C in the dark. All standard preparation is recorded in the TALs (LIMS) system. Solutions are prepared every 2-weeks (1-week for the gases, 2-CEVE, Vinyl Acetate, Acrolein and Nitriles).

Purgeable Spike Standard Mixes
ACCUSTANDARD, o2si and Absolute Standard

VOC LIQUID MIXTURE
2000 ug/mL in MeOH

1,1,1,2-Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,1-Dichloropropylene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
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-Chlorotoluene
4-Chlorotoluene
4-Isopropyltoluene
Benzene
Bromobenzene
Bromoform
Carbon tetrachloride
Chlorobenzene
Chlorodibromomethane
Chloroform
cis-1,2-Dichloroethylene
cis-1,3-Dichloropropylene
Dibromomethane
Dichlorobromomethane
Dichloromethane
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
m-Xylene
n-Butylbenzene
n-Propylbenzene
Naphthalene
o-Xylene
p-Xylene
sec-Butylbenzene
Styrene
tert-Butylbenzene
Tetrachloroethylene
Toluene
trans-1,2-Dichloroethylene
trans-1,3-Dichloropropylene
Trichloroethylene

Bromochloromethane
2000 ug/mL in MeOH

Heptane
2000 ug/mL in MeOH

MTBE
2000 ug/mL in MeOH

1,3,5-Trichlorobenzene
2000 ug/mL in MeOH

Clorohexane
1000 ug/mL in MeOH

Ethyl Ether
1000 ug/mL in MeOH

Hexane
1000 ug/mL in MeOH

Trichlorotrifluoroethane
2000 ug/mL in MeOH

Volatile Organic Compound Gas Spike
2000 ug/mL in MeOH
Chloroethane
Methyl Bromide (Bromomethane)
Methyl chloride (Chloromethane)
Trichlorofluoromethane
Dichlorodifluoromethane
Vinyl chloride

Volatile Mix Additional Spike Compounds
2000 ug/mL in MeOH
Acetone
2-Hexanone
Methyl ethyl ketone
4- Methyl-2-pentanone
Carbon Disulfide
Vinyl Acetate
2-Chloroethylvinylether
Iodomethane

8260 Custom Solution
2000 ug/mL in MeOH
Methyl Acetate
Methyl Cyclohexane
Cyclohexane

Dichlorofluoromethane
1000 ug/mL in MeOH

5.5 Stock Spike Solution

The spike compounds are obtained as solutions from a second source (i.e., Accustandard) in 1.5-2.0 mL ampules. These are listed on the previous page. A different analyst than the one who prepared the calibration solutions usually prepares spike solutions. These are stored according to manufacturer's recommended storage conditions. Neat standards are kept for a period of 1-year un-opened or the manufacturer's expiration date. Once opened, the stock may be used for 3-months.

The matrix spike solutions are prepared as follows:

5.5.1 VOC Spike

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
2000 ppm VOC Liquid Spike	50	Dilute to 2 mL	50 ppm each component
2000 ppm 8260 Additional Spike	50		
1000 ppm 1,3,5-Trichlorobenzene Spike	100		
2000 ppm Tetrahydrafuran	50		

5.5.2 Gas Spike

Mix	Volume (uL)	MeOH	Concentration
2000 ppm Gas Spike	50	Dilute to 2 mLs	50 ppm each component
1000 ppm Dichlorofluoromethane	100		

5.5.3 Additional Spike Compound Mix

Compound	Volume[1] (uL)	MeOH	Concentration
2000 ppm Bromochloromethane	50	Dilute to 2 mL	50 ppm each component
1000 ppm Ethyl Ether	100		
1000 ppm Chlorohexane	100		
1000 ppm Hexane	100		
2000 ppm MTBE	50		
1000 ppm Heptane	100		
1000 ppm Trichlorotrifluoroethane	100		

Compound	Volume (uL)	MeOH	Concentration
2000 ppm 8260 Custom Solution	50		

For waters, addition of 5 uL of each solution results in all spike compounds at 50 ppb.
For soils, addition of 5 uL of each solution results in all compounds at 50 ppb.

These solutions are stored at ~ -10°C in several 1.5-2.0 mL amber Teflon-lined screw-capped vials. All standard preparation is recorded in the TALs (LIMS) system. Working matrix spike solutions have a 2-week/1-week (Gases) expiration date or until low recoveries of the matrix spike compounds indicate a new solution is needed. See above for label information.

5.6 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Stock	Amount	MeOH	Concentration
2000 ppm BFB	25 uL	Dilute to 2 mLs	25 ppm

- Life of Standard: This stock can be kept for a period of 1-year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN. Once opened, it is used for a period of 6-months.
- Storage Requirements: The standard is stored at ~ –10°C in the dark

Addition of 2 uL to 5 mLs results in a concentration of 50 ng/5 mLs.
All preparation is recorded in the TALs (LIMS) system. All labels are completed as above.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

6.0 CALIBRATION

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

1. If an AUTOTUNE is to be performed, continue below. If not, skip to step 6. An autotune is not run before every initial calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing initial calibration is being met.

2. The Chemstation software has a menu driven tune program. Begin the autotune program. Key masses are 69, 219 and 502.

3. Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

4. The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e., the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce a target abundance for mass 69 (varies depending on the tune program and instrument). The operator may plan on having to increase this by 100-200 to achieve normal analysis sensitivity (depends on the tune program and the instrument).

5. Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

6. If an AUTOTUNE has just been performed, continue here. If not, skip to step 9. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

7. Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	40-60%
219	50-70%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 to 0.550 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

8. A hardcopy the profile scan is desirable. This can be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the initial calibration.

Save the changes to the appropriate Tune File. Exit the program.

9. If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. A hardcopy of profile scan can be printed and filed. Save the changes and exit the program.

6.2 BFB Analysis

Once the instrument is tuned, 50 ng of 4-Bromofluorobenzene must meet criteria. The BFB can be purged. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	15-40% of mass 95
75	30-60% of mass 95
95	Base Peak, 100% rel. abund.
96	5 - 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 - 9% of mass 174
176	>95% but <101% of mass 174
177	5 - 9% of mass 176

The manner of acquiring the mass spectrum of BFB can be performed in only two specific ways with Chrom software:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is performed and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. This procedure of averaging and subtracting is performed automatically by the Chrom software and evaluated by the criteria in the table below. If this procedure does not meet the acceptance criteria, then the analyst will use the following procedure:
- One scan of the BFB peak is acquired and a background peak is subtracted and evaluated by Chrom software using the criteria above. This procedure is performed manually by the analyst. No other manner of BFB acquisition and evaluation can be utilized.

The BFB is analyzed by one of the methods in Attachment 1. **(Method parameters listed in the appendices are examples only. This statement applies to all references made to these methods).** Typical concentrator conditions also appear in Attachment 1. The EM voltage may be 100-200 volts above autotune. The abundances of the designated masses above MUST meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. BFB analysis is completed about every 12-hours of analysis.

6.3 Description of Initial Calibration

An initial calibration may be completed:

- as needed - continuing calibration can not be met
- after a source cleaning and/or column change or any time a major repair or change has occurred with the instrument that affects calibration where a new calibration is indicated.

Confirm that the GC/MSD is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.
- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

6.4 Initial Calibration

Each calibration standard is analyzed according to one of the methods in Attachment 1. These are examples. The actual number of points in the calibration is determined by the calibration and acceptance criteria table (Attachment 4). The EM voltage may be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill ten 5-mL luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5-mL confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards. Following the guides found in the Attachment 1, add the appropriate amount of standards and methanol.

Immediately add the standards to a clean 40-mL vial. Following the method parameters outlined in Attachment 1, analyze the 50 ppb standard. A normal standard will appear very similar to the ones in Figures 1 and 2. Quantitate the standard against the appropriate method file. Sufficient areas for the first internal standard will vary somewhat between instruments. Acceptable areas should be based on maintaining sufficient sensitivity for poor responders without saturating the detector at the upper end of the calibration range. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

It is helpful to analyze a medium level standard first and assess the areas before continuing with the low/high level standards.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:
A_x = ion abundance for analyte
A_s = ion abundance for its internal standard
Q_s = concentration of its internal standard
Q_x = concentration of analyte
(Response Factors have no units)
The appropriate quant ion must be in the method file. A listing of the target compounds with their appropriate internal standards appears in Attachment 2. Confirm the presence of all targets and the separation of non-co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb standard.

The entire initial calibration shall be analyzed. If a point is found to be invalid due to an injection or instrument problem confined to that run, than the point may be repeated once only. If that point fails again a new ICAL must be performed. Removal of points for individual analytes from levels other than the highest or lowest level is not permitted in any event. Unused or replaced standards must have a clear explanation of why they were not used or why they were replaced. This explanation must be documented on the Corrective Action/Qualification Report bound with the run log.

When a standard is analyzed and processed as part of the initial calibration the RF's are automatically updated in the daily method. After all initial calibration standards are processed, checked and confirmed as being accurate and passing method criteria, the initial calibration is locked and set as the most recent. This ensures that the correct initial calibration is used for each ensuing continuing calibration check. A hardcopy of the calibration report is generated. All method criteria are assessed for compliance. Confirm that:

1) all CCC's are below 30% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2) the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

Calibration curves are evaluated following the "Evaluation and Acceptance Criteria" table (Attachment 4). For all compounds in the initial calibration with a %RSD > 15.0%, calibration curves of area ratio versus concentration using a first or higher order regression curve of the calibration curve points will be performed. Weighted linear curves may be used. Quadratic curves may only be used after evaluation of the curve plot and Supervisor approval (Note: The state of South Carolina does not allow the use of quadratic curves, therefore, in those cases, only the weighted linear option is allowed).

Method 8000B/8260B specifies a minimum coefficient of determination (R^2) of 0.990. The methods also specify a minimum of 5 calibration points for a linear model and a minimum of 6 calibration points for a higher order regression. The laboratory, in order to meet AFCEE requirements, will analyze a minimum number of points to satisfy both the SW846 and AFCEE. All efforts will be made to meet the minimum COD of 0.990. However, there are some compounds that historically present a problem meeting this requirement (See attachment 9 for a list of poor purgers). These compounds are usually those listed in the analyte table of Method 8260B with qualifying remarks. Many of these have various known issues that would effect reproducibility (i.e., Acetone qualifier pp = poor purger). These typically include many of the Appendix IX compounds as well. The laboratory will take minimal action for these compounds.

The preparation instructions noted above will be modified to include the necessary calibration levels. These instructions are for guidance only and may change as needed.

An example of an acceptable initial calibration appears in Attachment 2. The BFB tune, and all standard raw data are filed. Each instrument has its own initial calibration.

Each time a new initial calibration is required, the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds and venders). The ICV must meet 30%D for all compounds or corrective action must be taken. Some client's require 20%D. For South Carolina, all compounds must meet 30%D unless there are compounds identified as poor purgers in the SOP. The poor purgers should still meet 40%D. South Carolina does not allow the use of marginal exceedances. Multiple ICV's without documented corrective action or valid reason for doing so, is not acceptable.

NOTE: The actual number of points in the calibration and the low point in the calibration may vary with client and project need. Clients may have additional requirements, which would be covered in a client-specific or regulatory/agency QAPP.

6.5 Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

If time remains after the initial calibration, and the 50 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12-hour tune limit. The samples are quantitated against the average RF or appropriate as per method. See later sections describing calculations.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb standard following the procedure outlined above. Confirm Form 7 that:

1. all CCC's are below 20% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2. the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

NOTE: Method 8260B stipulates that if the CCC's are not part of the analyte list or a short list of compounds is being analyzed, then all compounds being reported must be < 20% drift.

If continuing calibration can not be met, either new standards and/or a new calibration are needed. Multiple CCV's without documented corrective action or valid reason for doing so is not acceptable. Since a CCV can fail for many reasons it is important to determine why it failed or where the problem originated (reagents, autosampler, concentrator, GC, Mass spectrometer).
If the initial CCV fails method criteria, try and determine why it failed:

- need new standards?
- manual tune needs adjusting?
- carry-over issues?
- low or high internal standard responses?

Take appropriate actions to correct the problem. Document corrective actions on the corrective action/qualification report. Re-analyze the CCV. If the second CCV fails method criteria, compare the two standards:

- Did corrective action help?
- Are there any trends between the two standards?
- Baseline rise or ghost peaks?
- Any indication of leak or gas contamination?
- Does further maintenance need to be performed?
  - Autosampler
    - replace needle?
    - Check flows and temperatures
  - Concentrator
    - Change trap?
    - Check flows and temperatures
  - GC
    - liner/seal need to be replaced or cleaned?
    - Check flows/temperatures
  - Mass spectrometer
    - Instrument need to be cleaned?
    - Mutltiplier and filaments stable?

TNI standard EL-VM4-2009 Mod 4 section 1.7.2 states: " If the continuing calibration verification results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial calibration shall be performed". Immediately running an ICAL without correcting the problem would not be productive. It could result in analyzing samples on a poor ICAL or on an instrument that is not functioning correctly. If two CCV's fail and corrective action was performed then, two acceptable consecutive CCV's must be obtained to prove the corrective action fixed the problem. If an acceptable CCV can not be obtained and the instrument appears to be stable then a new initial calibration should be analyzed.

All internal standard areas and retention times are assessed immediately after calibration. Areas and times compared to the mid point of the initial calibration. Internal standard areas should not deviate by a factor of two or the retention times should not deviate by > 30 s. If the situation occurs, appropriate action is taken and the standard re-analyzed. All corrective action and return to control are documented in the Corrective Action section in the analysis logbook for the appropriate instrument.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Control is accomplished through:
1) daily tuning and calibration checks and
2) preparation QC traceable through individual batches.

7.1.1 Initial Calibration

PFTBA BFB TUNE	Prior to Initial Cal	*limits in Section 6.2
200 \ 150 | 100 | 50 | 20 | 5 | 2 | 1 | 0.5 /	Initial Cal need dependent on situation.	*limits in Section 6.4

NOTE: As stated, the actual number of points in the calibration and the low point in the calibration may vary with client and project need. Minimum number of points for AFCEE and/or 3rd Edition SW-846 may be 6 or 7 depending on matrix. Other clients may have additional requirements, which would be covered in a client-specific QAPP.

7.1.2 Method Blank (MB)

Prior to any analysis, the reagent water and the instrument must be shown to be free of interference's and target compounds.

A 5 mL portion of reagent water is analyzed using one of the methods in Attachment 1. Internal Standards (ISS), Surrogates (SSS) and methanol will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. All target compounds must be less than the quantitation limit (See Section 2.0). Once the MB analysis is complete and acceptable, analysis can proceed.

7.1.3 Daily Analysis

BFB	Prior to continuing	* See above calibration
Daily Calibration Standard	Prior to samples	* See Section 6.5

Prep QC	Frequency
MB	Prior to analysis
LCS[1]	1 per analysis batch
MS/MSDs[2]	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
Samples *	

***Any given 12-hour period contains a tune, standard, blank and LCS. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.**

[1] LCS Duplicate (LCD) is performed when insufficient sample is available for an MS/MSD.
[2] The sample selection for MS/MSD, if not specified by the client on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Resource Conservation and Recovery Act (RCRA).

Matrix	SW-846
soils	14-days
Preserved waters	14-days
Non-preserved waters	7-days

All samples received for volatile analysis are refrigerated upon receipt at 4±2°C. Refrigeration is the only preservative for 5030 soil samples, while 5035 soils can be preserved with sodium bisulfate or frozen. Water samples are preserved with 3 drops of 36% HCl to a pH <2. Water samples marked as un-preserved are analyzed within 7-days.

7.3 Sample Preparation / Screening /Analysis

Once the samples are logged into the TALs (LIMS) database upon receipt, a paperwork trail is initiated. The Supervisor or Analyst prints and prepares the necessary information (Sample Tracking Sheets) and places it in the appropriate file bin in the GC/MS VOA lab. The analysts take this information and subsequently screen the associated samples. Samples are screened by MSD prior to analysis. The actual screening procedures vary due to sample appearance, sample matrix, client history and analytical method. Once the samples are screened, the paperwork is transferred to a second file appropriately labeled. This file contains information about samples that have been screened but need to be reviewed. Once screened, an 'X' is placed on top of the vial, indicating both that the sample has been screened, and that the particular vial can not be used for subsequent analysis. The screened analysis can be reviewed on screen or hard-copy, as all screening data is collected and stored on the data system as with all GC/MS analyses. Upon review, the analyst makes decisions concerning the screen and indicates if an initial dilution is required. This information is physically recorded on the paperwork. Once reviewed, the paperwork is then placed in appropriate files that are broken down by matrix and method. The samples are now ready to be analyzed.

7.3.1 Waters

Allow samples and standards to come to ambient temperature. Observe all vials of sample and confirm the absence of any air bubbles. If a vial has an air bubble do not use that vial and notify the Supervisor of the situation. Make sure the vial used for screening is properly marked and do not use this vial for analysis.

Remove the plunger from a 5 mL luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger. The pH of all samples is verified at time of analysis. If the is pH < 2, a check-mark is placed in the appropriate column on the sample tracking sheet. If the pH > 2, the actual estimated pH is written in the same column. pH checks and verification of hold-times are documented on the review form. Samples lacking preservation may be noted in the case narrative. Invert the syringe, and adjust the volume to 5 mLs. Confirm the absence of all air bubbles.

Add IS, SS and methanol to the 5 mL syringe. Initial concentrations of both internal standard and surrogate solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Immediately add the sample to a clean 40 mL vial using the method described in attachment 2 to analyze the sample.

If a batch is going to be analyzed, which is usually the case, load all samples, including all batch QC prepped in the same sequence and manner as the samples, following the procedure above. After the batch is loaded, replace all samples and standards back in storage. Appropriate documentation is made on the ICOC page.

If a dilution is required as indicated from the screening results, the following guidelines are followed. If the dilution is > 1/100 (250 uL of sample) an initial dilution is made into a volumetric flask. If serial dilutions are required, no less than 1 mL is taken for further dilutions. The final sample aliquot taken for analysis from the volumetric is no less than 250 uL. If the dilution is < 1/100, the appropriate sample amount is added directly to the 5 mL syringe.

Opened sample vials are used only once unless: (a) any necessary dilutions/reruns are done the same day or (b) there are no other vials for that sample.

7.3.2 Soils

As some clients still request method 5030 at the present time, soils are still being analyzed as indicated below. As clients convert to Method 5035 completely, this section will be removed.

Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a problem is noted, contact the QC department.

Allow samples and standards to come to ambient temperature.

Weight out 5 grams of the sample into a clean 40-mL vial. Add 5-mL reagent water into vial. IS/SS and methanol will be added though the septum. The autosampler will add an additional 10-mLs of reagent water. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Using the methods described in Attachment 1, analyze the sample. All soil samples are analyzed with a heated purge (40°C).

If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, including all batch QC prepped in the same sequence and manner as the samples, replace all samples and standards to their appropriate storage location. Documentation is made on the ICOC page.

Any sample that based on screening results/historical data has shown to contain high concentrations of compounds is analyzed at an initial dilution. Any sample that after the initial run contains targets above the calibration range is diluted to accurately quantitate those compounds. If an initial analysis over-diluted the given sample it is re-analyzed as a low level soil. If the low-level analysis contains compounds above the calibration range, and the same compounds are within range in the dilution, both sets of data may be reported to the client.

If a 1/2 or 1/5 dilution is required, 2.5g/1.0g of sample is weighed into the purge vessel.

7.3.3 Medium-Level Soil Extracts

If a larger dilution is required, a medium-level soil extract is prepared as follows. Five grams of sample is weighed into a tarred vial. Five 5-mLs of MeOH is added to the vial and the vial sealed. After the 24-48 hour contact time, the MeOH portion is decanted and stored in a 1-1.5 mL Teflon-lined screw-capped vial for storage. A portion of the extract (100 uL maximum) is taken for analysis. ISS and SSS will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Serial dilutions, if needed, are made from the extract and appropriate amounts taken for analysis.

If the sample upon which a medium-level prep as been performed also required an MS/MSD, the appropriate amount of MS solution is also added.

All samples prepared in this manner will be analyzed against a medium-level soil curve. The standards, blanks and LCS samples will contain 100 uL MeOH. The curve will be at ambient temperature.

NOTE: Some soils are analyzed initially at low levels due to increasing client requests for lower reporting limits. The same samples may then require large dilutions to bring compounds into the calibration range of the instrument. Some compounds, most notably the ketones, have very different responses when heated versus non-heated, despite the sample matrix. Traditionally, the lab heats soils. Therefore, the match between original analyses and dilutions for compounds such as these may not appear to correlate.

Dilution	Sample Weight	Vol. MeOH (1/2.5) Extract
1/2	2.5 grams	---
1/5	1.0 gram	---
1/50	5 grams / 5 mLs	100 uL
1/250	5 grams / 5 mLs	20 uL
1/500	5 grams / 5 mLs	10 uL

Using those parameters in Attachment 1, analyze all samples in the batch.

Sample vials/jars are only used once unless: (a) any dilutions/reruns are analyzed the same day or (b) there is only one jar for analysis.

7.3.4 Method 5035

NOTE: ICAL Standards are prepared with 5 mL milli-Q water.

Samples for low level VOA soil analysis may be received at the lab in one of three manners: First, as replicate 5 gram core samples in 40 mL vials containing organic free water and frozen within 48 hours of collection. Secondly, as replicate 5 gram core samples in 40 mL vials containing a Sodium Bisulfate preservative solution (refer to USP-5035 for collection/preservation). Thirdly, unpreserved 5 gram core samples may be received in Encore containers. These core samples must be placed in organic free water and frozen within 48-hours of collection. This time requirement is currently under review by appropriate regulatory agencies and may be extended beyond the 48-hours. Until such time, the laboratory will endeavor to "fix" the sample cores in preservative within 48-hours of collection. The laboratory may receive replicate 5 gram soil cores to be used for reanalysis if needed.

In addition to low level samples, an additional soil aliquot should be received for use as a screen and possible use as a mid-level extraction/analysis. This additional core must also be fixed in MeOH within 48-hours of collection. The amount of MeOH added must closely correspond to a soil to solvent ratio of 1:1. TestAmerica may adjust the methanol levels of any sample prepared in the laboratory from encore plugs with a corresponding 1:1 ratio of MeOH to soil. Any sample prepared in the field and fixed in methanol prior to arrival to the laboratory will not be adjusted but an NCM will document the discrepancies. Though not specified in the method, TestAmerica will pursue a goal of removing the MeOH from the soil within 24-48 hours after the initial extraction. A portion of the MeOH be removed and placed in an amber 1.5 - 2.0 mL Teflon-lined screw-capped vial for storage. This time limit should standardize the amount of time the MeOH comes in contact with the sample.

MeOH extracts of soils will be analyzed as stated above at ambient-temperature against a medium-level soil initial calibration. All surrogate and internal standard solutions will be added at time of analysis.

Low level soils will be analyzed using the Closed Purge and Trap Auto Sampler System. Surrogate and internal standard solutions will be added at the time of analysis through the septum by either a small gauge (10uL) syringe or automatically by the Archon autosampler. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and the spike and surrogate tables provided in this SOP. The concentration of the solution and amounts spiked may vary depending on the precision obtained with a given solution/volume combination. However, the final concentrations of such compounds in the samples will follow the same guidelines as previously stated in this SOP for all other samples.

As with the internal standard and surrogate, all QC spike solutions must also be added to the closed sample container. This is accomplished by the addition of the spike solutions through the septum with a small gauge (10 uL) syringe just prior to the sample being placed on the instrument for analysis.

7.3.5 Drum/Waste Samples

- Non-MeOH Miscible
- MeOH Miscible

These samples are normally treated as medium level soils or waste dilutions. Waste dilutions normally consist of 5 grams of sample to 5 mLs of MeOH. If the sample is non-miscible with methanol, mix the sample and allow sample to separate. Draw off the methanol into a screw top vial. Use a portion of this methanol extract to screen. Continue to analyze the sample as high a high level methanol extract at a dilution based on the screening result.

If the sample is miscible with methanol, notify the PM and provide a detailed description of sample matrix and any matrix issues. The sample will automatically be given a 1:2 dilution factor, as a result of its miscibility with the methanol. Screen the sample and continue to analyze by the high level methanol extract procedure based on the screening results. Both ISS and SSS are added prior to analysis. The laboratory can also pre-spike the surrogates and spike compounds if client-specified to do so. In many cases, due to the high level dilutions required, the surrogates and matrix spike compounds may be diluted out. Therefore, unless specifically instructed, both solutions will be added at the time of analysis.

Drum/waste samples that are biphasic in nature require a discussion with the PM and client in order to determine how the samples will be prepared and analyzed (one phase, both phases?). The preparation and analysis of the sample will be documented.

7.4 Preventive Maintenance

Instrumental maintenance can be categorized as daily and "as required". Required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. A description of the situation, actions taken and follow-up must be documented in the instrument maintenance logbook on the day of maintenance and initialed / dated. An example Maintenance logbook page can be found in Attachment 3. Maintenance logs are to be peer reviewed by a qualified analyst other than the analyst who recorded the maintenance in the logbook. The maintenance logs should be reviewed on a monthly basis.

7.4.1 Daily Maintenance

The most routinely performed maintenance includes:

- Purge-line or sample transfer line rinses within the concentrator and vial autosampler
- Analysis of blanks after high level samples
- GC oven bake after high level samples

The Corrective Action/Qualification Report for GC/MS VOA is used to document that the instrument Preventive Maintenance as described in this SOP has been performed.

7.4.2 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Concentrator, or MS related.

7.4.2.1 GC Related

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap
- Replace liners

7.4.2.2 MS Related

- clean source/rods and anything associated with that activity
- replace electron multiplier
- change filaments

7.4.2.3 Concentrator Related

- change transfer line; clean transfer line
- replace trap; condition new trap
- run 20 ug/L Bromoform standard to check for formation of Chloromethane and Bromomethane.
- refurbish Concentrator
- check purge pressure and flow rate
- analysis of position blanks after high-level samples
- change bulk head fitting

7.4.2.4 Autosampler Related

- change sparge needle
- change pencil filters
- flush standard pickups
- calibrate standard valve
- run vial position calibration
- clean transfer rods
- oil bearings

7.5 Documentation/Tracking of Sample Analyses

7.5.1 The GC/MS VOA lab employs several forms that serve both a tracking and review function. The Sample Tracking Sheet is filled out for each job. It contains information the analyst needs as for method, QC requirements, special reporting requirements, screening results, methanol lot # etc.., in addition for space to track the analysis of every single sample in the job and the outcome of that analysis. The Sample Tracking Sheet can be found in Attachment 3.

7.5.2 In addition, all samples logged into the department appear on back-logs ordered by both Hold Time and Due Date. The back-logs are utilized by the analysts when making decisions as to methods and analyses that are needed for the day. As samples are analyzed and reviewed, the back-logs are updated to reflect those samples completely analyzed, those requiring dilutions and re-analyses.

7.5.3 In addition, an instrument sequence log is printed, reviewed, signed and bound in a log daily for each instrument.

7.6 Archival of Data

Data will be available on the local server for at least 1 year. No data is removed from the system until it has been archived on at least one full back up and a number of differential back-ups.

Data is transferred to servers after each sample run. The servers are backed up to a local backup server each night. The local backup server is backed-up off-site daily. Data can be restored from the backup server. Back-ups are the responsibility of the IT Manager. Clarification of procedures can be found in the *UP-IS-014,* TestAmerica Chicago IT Procedures and Processes SOP.

7.7 Removal of Data

There is a substantial amount of space available to both BNAs and VOAs on the current data system. Data older than approximately six months is zipped and remains on the system. At the IT Manager's, Technical Manager's or GCMS Supervisor's discretion, previously archived data is purged from the system.

8.0 QUALITY CONTROL

8.1 QC Summary

The department will review the quality controls as follows:

8.1.1 Method Blank (MB) / Laboratory Control Standard (LCS)

At least one MB and LCS will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media.

The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy and precision.

8.1.2 Accuracy

Accuracy will be measured by the percent recovery (%R) of the LCS. Method 8260B refers to Method 8000 for guidance on initial demonstration performance criteria. Guidelines for LCS/LCSD and MS/MSD accuracy limits can also be found in Method 8000. The laboratory's current in-house statistical limits can be found in Table 1. The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project, or state certifications etc. This information is transmitted to the bench via the COC, kick-off meetings, tech profiles, job note or NCM etc.., and indicated on one of the forms used at the bench. In-house generated limits are subject to change.

8.1.3 Precision

Precision will be measured by the reproducibility of the LCS and will be calculated as Relative Percent Difference (RPD). Current limits are listed in Table 1. RPD's are not used to assess bench level Corrective Action.

8.1.4 Surrogates

Surrogate Compounds will be added to every sample to measure performance of the analysis. Method guidance limits are listed in Attachment 2. In-house statistical limits are listed in Table 1. Guidelines for the generation of statistical surrogate limits can be found in Method 8000. As with LCS samples, surrogate recovery limits may vary with client, QAP, project, etc.., and the information transmitted to the bench in the same manner.

8.1.5 QC Charting/Generation of Statistical Limits

Precision and accuracy are monitored using LCS data. Review of QC Charts and generation of in-house statistical criteria, including surrogate limits, is completed on an annual basis. Additional data may be added at QA/QC discretion during the year for other purposes. Spike levels are 50 ppb. Only routine compounds are spiked and should be representative of the whole. The more non-routine compounds are not part of the spiking solutions. Other limitations (availability of second source) may also prevent adding these to spiking solutions.

8.2 Corrective Actions

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst must address the following issues as described below in the individual sections.

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the supervisor, project manager, QC personnel or other qualified personnel prior to release of data associated with the problem.

Corrective Actions are documented on the Corrective Action/Qualification Report included in the instrument logbook. In addition, a sample tracking form, specific to a unique job, is attached to the sample tracking documentation. The corrective action/qualification report and sample tracking form are used to note all out-of-control events, the actions taken to try and correct the problem, the return to control.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

8.2.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune-related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Corrective action for BFB analysis is documented on the corrective action/qualification report in the logbook.

8.2.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e., a leak
- check the response factors - is one concentration level very high or low? re-analyze
- how old are the standards?

All calibration criteria must be met (Section 6.4). If the ICAL does not meet specified criteria, at minimum, the appropriate levels must be re-analyzed. If necessary, new standards should be prepared and the levels re-analyzed. During analysis of an initial calibration, documentation of the re-analyses of specific levels is required. See previous section outlining CA for minimum COD values as well. Refer to the TestAmerica Corporate Policy, CA-T-P-002, *Selection of Calibration Points* (Attachment 7), for further guidance.

8.2.3 ICV/Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
- is overall sensitivity low?
- excessive background?
- how old is the standard?
- need a new 5-point?
- has the tune shifted?

Compare the relative abundances of 69, 131 and 219 from that day's manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in. Certain compounds will help indicate what the problem is.

All calibration criteria must be met (Section 6.5). If the ICV/CCAL does not meet specified criteria, at minimum the standard should be re-analyzed. A new standard may be prepared and then re-analyzed. If necessary, a new ICAL must be run. All corrective action taken for CCAL's must be recorded on the corrective action/qualification report and included in the logbook. Multiple ICV/CCVs without documented corrective action or valid reason for doing so, is not acceptable.

8.2.4 Method Blank (MB)

If the MB is/appears to be contaminated, re-analyze. If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e., replace trap) it is documented in the Maintenance logbook. All corrective action taken for Method Blanks must be recorded on the corrective action/qualification report and included in the logbook. Under extenuating circumstances, if analysis continues, qualification must be made as to the positive hits above the RL for the compounds in question. Any associated samples analyzed in the tune must be noted. Any samples containing positive hits must be noted. IF, the samples containing positive hits can not be re-analyzed (i.e., past hold-time), the positive hits are flagged with "B" and the situation and data noted and qualified in a case narrative and/or Non-Conformance Memo (NCM).

8.2.5 Surrogates

All surrogate recoveries are calculated. If ANY surrogates are outside limits in the MB, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e., trap replacement, clean instrument).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution, if there is reasonable expectation that a high concentration of a target compound is causing a matrix effect. If the surrogate(s) is/are still outside limits, a matrix effect is demonstrated and both reports are submitted. Depending on the client, the best result may be reported and the other result narrated. If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e., capacity hold-times preclude re-analyses within hold-time), both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD were performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

Surrogate corrective action is documented on the sample tracking form for samples.

8.2.6 Laboratory Control Sample (LCS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project etc. The in-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and positive detected of compounds outside limits. The LCS limits are based on the mean recovery +/- 3 standard deviations; therefore, it is statistically quite likely that there will be exceedances of the limits for a few analytes when a large number of compounds are included in the LCS. Therefore a number of individual analytes are allowed to exceed the LCS control limit before the LCS as a whole is considered to have failed. The control limit for a marginal exceedance is mean +/- 4 standard deviations which results in a larger range of +/-10% outside the in-house generated limits. The number of marginal exceedances allowed is based on DOD guidance:

Number of analytes in LCS	Number of marginal exceedances allowed
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

- For QAP's specifying five compounds-- All five compounds must be within limits for analysis to proceed. The LCS samples may be re-analyzed. New spike solutions may be prepared. Or new standards or CCAL's may be analyzed. All corrective action and return to control must be documented at the time on the corrective action/qualification report and included in the logbook.The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, the above corrective action and required documentation apply.

- For full list spikes, all recoveries are assessed, although no immediate corrective action may be required if within the marginal excedance. If the recoveries are low, in general another LCS may be re-analyzed. The spike solution and standard may be verified for correct concentrations. However, no corrective action is absolutely required by the bench unless an error is discovered. The recoveries may or may not be documented in a NCM, however, they are noted on the review form.

- Although not strictly required to take immediate corrective action, the purpose of the full-spike is two-fold in that the bench should use it as an indicator of the status of the calibration standards, instrument conditions etc.., as well as a tool for data interpretation. Therefore, in keeping with good lab practice, the situation should be noted and assessed and any corrective action deemed necessary should be taken within a reasonable amount of time (Example: High recoveries on gases => new calibration standard may be needed).

- For South Carolina, all compounds in the LCS must meet 70-130% unless the compounds are identified as poor purgers in the SOP. The poor purgers should still be within 60-140%. South Carolina does not allow the use of mariginal exceedances.

8.2.7 Matrix Spikes (MS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project, state certifications etc. In-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and recoveries of same compounds in the associated LCS samples. The following guidelines are used:

(1) AFCEE or Specified Projects: All compounds are used for bench level control.

- If the MS exhibits recoveries outside limits, AFCEE requires it to be re-analyzed as the MSD. No further action is required. Documentation is required and association made to the LCS for those compounds in the NCM. See above specifications for associated LCS samples.

(2) QAP's etc., specifying 5 compounds.

- ALL 5 compounds are assessed. If recoveries are outside limits, the LCS is reviewed for those compounds. If the recoveries are within limits in the associated LCS samples, no further action is required. See above section concerning LCS corrective action for further information and action required for recoveries outside limits in LCS samples.

- The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, corrective action and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. The affected compounds may be compared to the same compounds in the associated LCS samples. See the above section for further information and action required for these compounds in the LCS samples. The recoveries may or may not be documented in the Job narrative, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

8.2.8 Internal Standard Policy

Method 8260 does not require re-analyses of samples for low internal standard areas. However, it is TestAmerica's policy to monitor areas and retention times, therefore, the following guidelines apply.

Situations requiring re-analyses :

- If ALL areas are outside limits the sample will be re-analyzed.
- Any sample that has a positive hit associated with any internal standard outside limits will be re-analyzed.
- If ANY surrogates are outside limits the sample will be re-analyzed.

Situations NOT requiring re-analyses :

- If all surrogates are within limits and there are no positive hits associated with those internal that are outside limits, the sample does not have to be re-analyzed. Situation should be addressed in a NCM.
- If all surrogates are within limits, but there is an obvious matrix effect occurring, even if positive hits are noted, the sample does not need to be re-analyzed. This decision will be approved by the supervisor. The situation should be addressed in a NCM.

- If there is historical evidence that shows a repeated pattern for a certain client and site, and this can be documented by reviewing past projects, the samples do not have to be re-analyzed. This decision will be approved by the supervisor and documented in a NCM.
- Corrective action for internal standard areas for samples is documented on the sample tracking form.

Any sample showing retention times outside windows will be re-analyzed. This is documented in the appropriate manner as in the preceding paragraph.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Chrom software produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 5 tentatively identified compound (TIC) search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present.

9.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system can be accomplished as follows:

WATERs: concentration (mg/L) = $\frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$

Where:
A_x = area of characteristic ion for target
I_s = concentration of internal standard (ng)
A_{is} = area of characteristic ion for int. std.
RF = response factor for target
DF = dilution factor (if any)

SOILs: concentration (mg/kg) = $\frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$

Where:
All variables are equal and
D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The target methods all contain calculations for waters and soils that allow automatic processing and calculations of concentrations to be completed. The user may enter some variables (Dilution Factor) and others are imported from LIMS. Sample prep info for VOA's is entered directly into LIMS. The sample volume is considered to be "constant" for calculation purposes. Less sample volume (in the case of waters) and soil weight (in the case of soils) are taken into account in the dilution factor entered by the user. For medium-level soils and waste/drum type samples medium level calculations are needed and actual weights are brought into LIMS.

NOTE: As noted previously, weights are recorded to 0.1 gram. It is SOP to weigh out 5.0 grams (or as appropriate for the dilution), however, to keep data entry and calculations simple. The same holds true for all water volumes.

9.1.2 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.1.3 Precision: $\% D = \frac{|B_1 - B_2|}{B_1} \times 100$

$$RPD = \frac{|B_1 - B_2|}{(B_1 + B_2)/2} \times 100$$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCS)

9.1.4 Modifications for 8260B quantitation

9.1.4.1 Initial Calibration Criteria

Methods 8000B/8260B require the use of linear or higher order calibration curves for those compounds exceeding 15%.

The following equations apply :

Linear Regression: $y = a_0 + a_1 * x$ Quadratic Curve: $y = a_o + (a1 * x) + (a2 * x^2)$
Weighted Linear Regression: $y = a_1 * x + a_0$

Where: $x = Area_{UNK}/Area_{ISTO}$
$y = Amount_{UNK}/Amount_{ISTO}$
a_1 = slope
a_0 = y-intercept

The equation for weighted linear regression follows the linear regression but introduces a weighting factor for the slope and y-intercept. The User manually enters the weighting factor into the method as: 1/Amt or $1/Amt^2$

Once the $Amount_{UNK}$ is solved, the value is adjusted for total solids, dilution factors etc.., to calculate a final concentration.

The quantitation of compounds using linear regressions and quadratic curves as performed automatically by the Target software has been confirmed to be accurate.

Method 8000B/8260B specifies a minimum COD(R^2). The corrective action regarding an initial calibration for method 8260B as it relates to the 0.990 correlation coefficient acceptance criteria is outlined. When a compound has a correlation coefficient less than 0.990, the occurrence is documented by the analyst in the Corrective Action section of the instrument's logbook. Any corrective action or data qualification is also documented on the corrective action/qualification report and included with the logbook. All corrective actions taken may include those listed below.

Samples may be analyzed against an initial calibration that have compounds with a correlation coefficient less than 0.990 and the corrective actions taken may also include some but not all of the following:

- The data for these samples may be reported without qualification if the compounds with a correlation coefficient less than 0.990 are not detected in the sample; therefore no further corrective action is required.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the samples may be reanalyzed against an initial calibration with an acceptable correlation coefficient and only the reanalysis will be reported on the sample. If this reanalysis occurs beyond analysis hold times then both analyses on the sample will be reported.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the decision to reanalyze or to reported the data without further corrective action is made on a case by case basis with the approval of the supervisor, the project manager and the client. The sample results may require qualification for this compound on the report and will be addressed in the case narrative.

9.1.4.2 Continuing Calibration Check

Prior to sample analysis a 50 ppb calibration check is completed. All minimum RF's must meet same limits. All CCC's must be less than 20% Drift as calculated below; the analysts may verify %DIFF and only calculate those that are close. (Error may only be made in favor of tighter control).

$$\%Drift = \frac{(Ci - Cc)}{Ci} \times 100$$

Where:
Ci = standard conc. (10/50)
Cc = measured conc. in cal check

9.1.5 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the Chrom processing software. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the MB is flagged with a "JB". Any TIC identified with a CAS number is also flagged with an "N", indicating that the ID was based on the mass spectra. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The Chrom system automatically calculates the actual concentration of the TIC's, including dilutions and total solids, once that information is retrieved from TALs (LIMS).

9.2 Operator Data Reduction/Review

The operator does on-screen review of all data and

- makes judgments concerning the "realness" of those target compounds found and
- makes judgments concerning the identification of the tentatively identified compounds
- modifies the output to produce a data package reflective of those decisions

9.2.1 Initial Review

The GC/MS VOA area uses two kinds of corrective action documentation. The first consists of the Batch Information section in Analyst Desktop in TALs (LIMs). This area in TALs contains a comment section to report out-of-control situations, corrective action and return-to-control for documenting problems related to general QC: tune, ICAL, CCAL, internal standard areas from CCAL to ICAL, and LCS samples. The second are the sample tracking forms that refer to a single job. These are used to record events, corrective actions and final actions for surrogates, internal standard areas, carry-over situations, analyses past tune time, MS/MSD data etc.., for each sample in the batch. These forms are attached to the other sample documentation that accompanies the job through analysis. Both may be used during initial review of the data. See Section 8.2 of this SOP for details on Corrective Action.

All data is initially reviewed on-screen. The review is both a QC review and a general review as described below.

- The MB contains no interferences or target compounds at the RL.
- ALL surrogates are in control in the blank. Surrogate limits are listed in Table 1;
- ALL surrogates in samples are in control;
- LCS recoveries meet the limits listed in Table 1. See Section 8.2 concerning compounds and limits for LCS samples. In-house limits have been generated and are in use.
- Internal standard areas and retention times are checked and meet guidelines. Limits are listed in Attachment 2. Additional guidelines can be found in Section 8.2.
- The sample does not require any further dilutions or analysis at a more concentrated level. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.
- Visually confirm complete integration for any large and/or saturated target compounds.
- The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc.).

9.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

1. "elution of component at the same relative retention time as the standard component."

The elution times should compare within +- 30 s. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

2. "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) should be present in the sample.

The relative intensities of the above ions should agree within ±20%, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions >10% in the sample but not present in the standard should be considered and accounted for.

3. Operator judgment. If a compound can not be verified by the above, but in the operators technical judgment the ID is correct, it is reported as such.

- Once all positive identification is made, the file is modified to reflect these decisions. At this time TIC's may also be reviewed and name. In each case where the file has been edited or manual integrations have taken place the operator must identify, initial and date the changes on the hardcopy (if such is generated).

9.2.3 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must identify, initial, and date the changes on the hardcopy report. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.
- Manual integrations shall follow the TestAmerica Corporate SOP for Manual Integrations (CA-Q-S-002 – Attachment 8). Example integrations and documentation are provided within this attachment.

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All manual integrations are reviewed, initialed and dated. For those clients requiring full data packages, spectra and Extracted Ion Chromatography Profiles (EICP) are printed for all manually integrated compounds. Manual integrations are documented in the case narrative and a Manual Integration Summary is included in the data package.

9.2.4 Identification of TIC's

In general, up to as many as 5 non-target compounds are tentatively identified by the data system and operator. Compounds with responses >10% of the nearest ISTD are identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within ±20%;
- Molecular ions in the reference should be in the samples;
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard;

- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds;
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds;
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

9.3 Final Review

9.3.1 Once (a) the analysis is determined to be acceptable and (b) the initial review and data reduction has occurred and (c) the analyst has entered sample prep info into TALs (LIMS), the following steps occur. The sample prep information, client ID information and some data applicable to fields in the forms is retrieved from TALs (LIMS).

9.3.2 All necessary forms are then generated using the TALs. The package is then assembled and ready for the first review. For level 2 data reports and similar deliverables other data may be generated for review purposes. In these cases, final packages with raw data and forms are not generated. Review of all reports and associated data is required regardless of data deliverable level.

9.3.3 Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 6) and sets the batch status in LIMs to 2nd level. The peer/supervisor review of the data is conducted by another individual who has been trained on the review process or by the department supervisor. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to lab complete. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

Note: The complete analysis scheme can be summarized below (Section 7.1.1 & 7.1.2) and in Attachment 5. The entire sample tracking system can be summarized in Attachment 5.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste from this procedure will enter the 'Flammable Vials' wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Estimated Quantitation Limits for Volatile Analytes; Laboratory Statistical Control Limits/ Surrogate Recovery Guidelines
Table 2. Characteristic Mass for Purgeable Organics Compounds

Figure 1. Example: Total Ion Chromatogram for 5 mL Purge Water
Figure 2. Example: Total Ion Chromatogram for 5 mL Purge Soil

Attachment 1. Example: Initial Calibration Guides; Method Listings; Concentrator Conditions; Flow Settings
Attachment 2. Example: Target and Internal Standards; Internal Standard Guidelines; Initial Calibration (Form 6)
Attachment 3. Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA-ICOC Form
Attachment 4. Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7)
Attachment 5. Example: Analysis and Sample Tracking Flowcharts
Attachment 6. Example: Data Review Checklist
Attachment 7. CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points
Attachment 8. CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations
Attachment 9. List of Poor Purging or Poorly Performing Compounds
Attachment 10 DOD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1; and Table F-4)

14.0 REVISION HISTORY

- Revision 23, was updated on 11/30/12
- Audit Response updates
- Section 4.1 was updated to reflect current Instrument Hardware / Software
- Section 5.2 and 5.3 were updated to include instrument reagent label requirements for Surrogates and Internal Standards
- General updated to standard sources and concentrations
- Section 6.5.2 was updated to reference specific language in the TNI Standard EL-V1M4-2009 section 1.7.2.
- Section 7.2 was updated to reflect current preservation and HTs

Table 1.

Example: Estimated Quantitation Limits for Volatile Analytes[a] Laboratory Statistical Control Limits and Surrogate Recovery Guidelines (040-001 to 040-012)

[a]Estimated Quantitation Limit (EQL) – The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 3 to 5 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following example information for further guidance on matrix-dependent EQLs.

[b]EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Note: the 8260B high level Methanol RL, LOD and MDL limits listed on the attached summary have a 50x dilution factor applied in the analytical batch.

[c]The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project, State Certifications etc. This information is transmitted to the bench via job notes and NCMs attached to the projects.

MDL S MSVOA_Low Default List	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,1,1,2-Tetrachloroethane	630-20-6	5	2.5	0.682	ug/Kg	70	120	30	70	120	30		
1,1,1-Trichloroethane	71-55-6	5	2.5	0.747	ug/Kg	66	127	30	66	127	30		
1,1,2,2-Tetrachloroethane	79-34-5	5	2.5	1.01	ug/Kg	70	125	30	70	125	30		
1,1,2-Trichloroethane	79-00-5	5	2.5	0.682	ug/Kg	70	120	30	70	120	30		
1,1-Dichloroethane	75-34-3	5	2.5	0.791	ug/Kg	67	120	30	67	120	30		
1,1-Dichloroethene	75-35-4	5	2.5	0.808	ug/Kg	53	122	30	53	122	30		
1,1-Dichloropropene	563-58-6	5	2.5	0.903	ug/Kg	70	120	30	70	120	30		
1,2,3-Trichlorobenzene	87-61-6	5	2.5	0.742	ug/Kg	64	128	30	64	128	30		
1,2,3-Trichloropropane	96-18-4	5	2.5	1.67	ug/Kg	70	126	30	70	126	30		
1,2,4-Trichlorobenzene	120-82-1	5	2.5	0.719	ug/Kg	64	120	30	64	120	30		
1,2,4-Trimethylbenzene	95-63-6	5	2.5	0.575	ug/Kg	75	120	30	75	120	30		
1,2-Dibromo-3-Chloropropane	96-12-8	5	2.5	1.68	ug/Kg	55	124	30	55	124	30		
1,2-Dibromoethane	106-93-4	5	2.5	0.941	ug/Kg	70	120	30	70	120	30		
Acrolein	107-02-8	400	200	39.8	ug/Kg	40	130	30	40	130	30		
1,2-Dichlorobenzene	95-50-1	5	2.5	0.603	ug/Kg	75	120	30	75	120	30		
1,2-Dichloroethane	107-06-2	5	2.5	0.741	ug/Kg	68	123	30	68	123	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							70	134
1,2-Dichloropropane	78-87-5	5	2.5	0.759	ug/Kg	70	120	30	70	120	30		
1,3,5-Trimethylbenzene	108-67-8	5	2.5	0.657	ug/Kg	75	120	30	75	120	30		
1,3-Dichlorobenzene	541-73-1	5	2.5	0.667	ug/Kg	70	120	30	70	120	30		
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	2.5	1	ug/Kg	41	120	30	41	120	30		
1,3-Dichloropropane	142-28-9	5	2.5	0.585	ug/Kg	70	120	30	70	120	30		
1,4-Dichlorobenzene	106-46-7	5	2.5	0.625	ug/Kg	75	120	30	75	120	30		
2,2-Dichloropropane	594-20-7	5	2.5	0.885	ug/Kg	61	127	30	61	127	30		
2-Chlorotoluene	95-49-8	5	2.5	0.618	ug/Kg	70	120	30	70	120	30		
2-Hexanone	591-78-6	5	2.5	1.44	ug/Kg	64	130	30	64	130	30		
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							70	122
Acetonitrile	75-05-8	80	40	20	ug/Kg	64	133	30	64	133	30		
4-Chlorotoluene	106-43-4	5	2.5	0.671	ug/Kg	70	120	30	70	120	30		
Acetone	67-64-1	5	2.5	2.16	ug/Kg	50	138	30	50	138	30		
Benzene	71-43-2	5	2.5	0.685	ug/Kg	70	120	30	70	120	30		
Bromobenzene	108-86-1	5	2.5	0.624	ug/Kg	75	120	30	75	120	30		
Bromochloromethane	74-97-5	5	2.5	0.966	ug/Kg	66	124	30	66	124	30		
Bromodichloromethane	75-27-4	5	2.5	0.861	ug/Kg	70	120	30	70	120	30		

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Acrylonitrile	107-13-1	80	40	11	ug/Kg	70	129	30	70	129	30		
Bromoform	75-25-2	5	2.5	1.15	ug/Kg	63	124	30	63	124	30		
Bromomethane	74-83-9	5	2.5	1.51	ug/Kg	50	150	30	50	150	30		
Carbon disulfide	75-15-0	5	2.5	0.747	ug/Kg	50	120	30	50	120	30		
Carbon tetrachloride	56-23-5	5	2.5	0.91	ug/Kg	63	124	30	63	124	30		
Chlorobenzene	108-90-7	5	2.5	0.507	ug/Kg	70	120	30	70	120	30		
Chloroethane	75-00-3	5	2.5	1.36	ug/Kg	50	150	30	50	150	30		
Vinyl acetate	108-05-4	5	2.5	0.786	ug/Kg	50	130	30	50	130	30		
Chloroform	67-66-3	5	2.5	0.575	ug/Kg	70	120	30	70	120	30		
Chloromethane	74-87-3	5	2.5	1.05	ug/Kg	50	130	30	50	130	30		
cis-1,2-Dichloroethene	156-59-2	5	2.5	0.707	ug/Kg	70	120	30	70	120	30		
cis-1,3-Dichloropropene	10061-01-5	5	2.5	0.656	ug/Kg	70	120	30	70	120	30		
Dibromochloromethane	124-48-1	5	2.5	0.87	ug/Kg	70	120	30	70	120	30		
Dibromofluoromethane	1868-53-7				ug/Kg							75	120
Dibromomethane	74-95-3	5	2.5	1	ug/Kg	70	120	30	70	120	30		
Dichlorodifluoromethane	75-71-8	5	2.5	1.43	ug/Kg	40	130	30	40	130	30		
Propionitrile	107-12-0	80	40	12.3	ug/Kg	69	134	30	69	134	30		
Ethylbenzene	100-41-4	5	2.5	1.01	ug/Kg	70	120	30	70	120	30		
Hexachlorobutadiene	87-68-3	5	2.5	1.14	ug/Kg	70	126	30	70	126	30		
Isopropylbenzene	98-82-8	5	2.5	0.586	ug/Kg	67	120	30	67	120	30		
m&p-Xylene	179601-23-1	10	5	1.23	ug/Kg	70	120	30	70	120	30		
Tetrahydrofuran	109-99-9	5	2.5	1.43	ug/Kg	70	125	30	70	125	30		
Methyl Ethyl Ketone	78-93-3	5	2.5	1.81	ug/Kg	58	133	30	58	133	30		
methyl isobutyl ketone	108-10-1	5	2.5	1.31	ug/Kg	68	126	30	68	126	30		
Methyl tert-butyl ether	1634-04-4	5	2.5	0.826	ug/Kg	62	123	30	62	123	30		
Methylene Chloride	75-09-2	5	2.5	1.35	ug/Kg	65	124	30	65	124	30		
Naphthalene	91-20-3	5	2.5	0.634	ug/Kg	67	128	30	67	128	30		
n-Butylbenzene	104-51-8	5	2.5	0.684	ug/Kg	70	120	30	70	120	30		
N-Propylbenzene	103-65-1	5	2.5	0.554	ug/Kg	70	120	30	70	120	30		
o-Xylene	95-47-6	5	2.5	0.453	ug/Kg	70	120	30	70	120	30		
p-Isopropyltoluene	99-87-6	5	2.5	0.54	ug/Kg	70	120	30	70	120	30		
sec-Butylbenzene	135-98-8	5	2.5	0.628	ug/Kg	70	120	30	70	120	30		
1,2-Dichloroethene, Total	540-59-0	5	2.5	1.21	ug/Kg	65	120	30	65	120	30		
Styrene	100-42-5	5	2.5	0.656	ug/Kg	75	120	30	75	120	30		
tert-Butylbenzene	98-06-6	5	2.5	0.597	ug/Kg	70	120	30	70	120	30		
Tetrachloroethene	127-18-4	5	2.5	0.764	ug/Kg	70	120	30	70	120	30		
Toluene	108-88-3	5	2.5	0.7	ug/Kg	70	120	30	70	120	30		

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Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	122
trans-1,2-Dichloroethene	156-60-5	5	2.5	0.688	ug/Kg	68	125	30	68	125	30		
trans-1,3-Dichloropropene	10061-02-6	5	2.5	0.896	ug/Kg	68	120	30	68	120	30		
Trichloroethene	79-01-6	5	2.5	0.825	ug/Kg	70	120	30	70	120	30		
Trichlorofluoromethane	75-69-4	5	2.5	0.778	ug/Kg	60	146	30	60	146	30		
Vinyl chloride	75-01-4	5	2.5	1.05	ug/Kg	61	137	30	61	137	30		
2-Chloroethyl vinyl ether	110-75-8	5	2.5	1.15	ug/Kg	50	130	30	50	130	30		
1-Chlorohexane	544-10-5	5	2.5	0.695	ug/Kg	60	130	30	60	130	30		

MDL S MSVOA_Low Default List	Closed System Purge and Trap	5035A_LP

(040-003)

MDL S MSVOA_Low APIX	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,3,5-Trichlorobenzene	108-70-3	5	2.5	0.947	ug/Kg	70	120	30	70	120	30		
2-Chloro-1,3-butadiene	126-99-8	5	2.5	1.24	ug/Kg								
2-Methylnaphthalene	91-57-6	5	2.5	1.33	ug/Kg								
2-Nitropropane	79-46-9	400	200	88.4	ug/Kg								
3-Chloropropene	107-05-1	10	5	2.42	ug/Kg								
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							70	122
Butadiene	106-99-0	5	2.5	1.28	ug/Kg								
Cyclohexane	110-82-7	5	2.5	1.47	ug/Kg	50	125	30	50	125	30		
Cyclohexanone	108-94-1	400	202	202	ug/Kg								
Ethyl acetate	141-78-6	50	25	12.8	ug/Kg								
Ethyl ether	60-29-7	5	2.5	0.955	ug/Kg	50	120	30	50	120	30		
Ethyl methacrylate	97-63-2	10	5	2.51	ug/Kg								
Hexane	110-54-3	5	2.5	1.22	ug/Kg	50	120	30	50	120	30		
Iodomethane	74-88-4	10	5	1.86	ug/Kg	57	134	30	57	134	30		
Isobutanol	78-83-1	400	200	106	ug/Kg								
Isopropyl ether	108-20-3	5	2.5	0.673	ug/Kg								
Methacrylonitrile	126-98-7	10	5	2.93	ug/Kg								
Methyl acetate	79-20-9	5	2.5	1.52	ug/Kg	60	140	30	60	140	30		
Methyl methacrylate	80-62-6	10	5	2.08	ug/Kg								
Methylcyclohexane	108-87-2	5	2.5	1.08	ug/Kg	60	140	30	60	140	30		
n-Butyl alcohol	71-36-3	400	200	102	ug/Kg								
Pentachloroethane	76-01-7	20	10	1.89	ug/Kg								
Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	122
trans-1,4-Dichloro-2-butene	110-57-6	10	5	2.17	ug/Kg								
2,2,4-Trimethylpentane	540-84-1	5	2.5	2.5	ug/Kg								

MDL S MSVOA_Low APIX	Closed System Purge and Trap	5035A_LP

(040-004)

MDL W GCMS VOA	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,1,1,2-Tetrachloroethane	630-20-6	1	0.5	0.25	ug/L	75	120	20	75	120	20		
1,1,1-Trichloroethane	71-55-6	1	0.5	0.2	ug/L	70	123	20	70	123	20		
1,1,2,2-Tetrachloroethane	79-34-5	1	0.5	0.23	ug/L	70	128	20	70	128	20		
1,1,2-Trichloroethane	79-00-5	1	0.5	0.28	ug/L	69	120	20	69	120	20		
1,1-Dichloroethane	75-34-3	1	0.5	0.19	ug/L	68	121	20	68	121	20		
1,1-Dichloroethene	75-35-4	1	0.5	0.31	ug/L	58	122	20	58	122	20		
1,1-Dichloropropene	563-58-6	1	0.5	0.34	ug/L	70	120	20	70	120	20		
Acrolein	107-02-8	100	50	10.9	ug/L	40	130	20	40	130	20		
1,2,3-Trichlorobenzene	87-61-6	1	0.5	0.24	ug/L	56	137	20	56	137	20		
1,2,3-Trichloropropane	96-18-4	1	0.6	0.45	ug/L	70	120	20	70	120	20		
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1	0.5	0.22	ug/L	46	120	20	46	120	20		
1,2,4-Trichlorobenzene	120-82-1	1	0.5	0.31	ug/L	65	121	20	65	121	20		
1,2,4-Trimethylbenzene	95-63-6	1	0.5	0.14	ug/L	75	121	20	75	121	20		
1,2-Dichloroethane	107-06-2	1	0.5	0.28	ug/L	69	120	20	69	120	20		
1,2-Dichlorobenzene	95-50-1	1	0.5	0.27	ug/L	75	120	20	75	120	20		
Acetonitrile	75-05-8	20	10	7.3	ug/L	60	131	20	60	131	20		
1,2-Dichloropropane	78-87-5	1	0.5	0.2	ug/L	70	120	20	70	120	20		
1,3,5-Trimethylbenzene	108-67-8	1	0.5	0.18	ug/L	75	123	20	75	123	20		
Acrylonitrile	107-13-1	20	10	2.6	ug/L	62	127	20	62	127	20		
1,3-Dichlorobenzene	541-73-1	1	0.5	0.15	ug/L	70	120	20	70	120	20		
1,3-Dichloropropane	142-28-9	1	0.5	0.13	ug/L	70	120	20	70	120	20		
1,4-Dichlorobenzene	106-46-7	1	0.5	0.15	ug/L	75	120	20	75	120	20		
2,2-Dichloropropane	594-20-7	1	0.5	0.32	ug/L	67	125	20	67	125	20		
Vinyl acetate	108-05-4	2	1	0.33	ug/L	50	130	20	50	130	20		
2-Hexanone	591-78-6	5	2.5	0.56	ug/L	55	144	20	55	144	20		
methyl isobutyl ketone	108-10-1	5	2.5	0.33	ug/L	59	135	20	59	135	20		
Acetone	67-64-1	5	2.5	1.29	ug/L	46	153	20	46	153	20		
Benzene	71-43-2	0.5	0.25	0.074	ug/L	70	120	20	70	120	20		
Propionitrile	107-12-0	20	10	3.5	ug/L	66	129	20	66	129	20		
Bromobenzene	108-86-1	1	0.5	0.25	ug/L	70	120	20	70	120	20		
Bromochloromethane	74-97-5	1	0.5	0.4	ug/L	67	122	20	67	122	20		
Tetrahydrofuran	109-99-9	5	2.5	1.2	ug/L	50	133	20	50	133	20		
Bromodichloromethane	75-27-4	1	0.5	0.17	ug/L	70	120	20	70	120	20		
Bromomethane	74-83-9	1	0.5	0.31	ug/L	50	150	20	50	150	20		

(500-40)

Bromoform	75-25-2	1	0.5	0.28	ug/L	70	125	20	70	125	20		
n-Butylbenzene	104-51-8	1	0.5	0.13	ug/L	75	120	20	75	120	20		
1,2-Dichloroethene, Total	540-59-0	2	1	0.58	ug/L	70	124	20	70	124	20		
cis-1,2-Dichloroethene	156-59-2	1	0.5	0.12	ug/L	70	120	20	70	120	20		
cis-1,3-Dichloropropene	10061-01-5	1	0.5	0.18	ug/L	70	120	20	70	120	20		
Carbon disulfide	75-15-0	5	2.5	0.43	ug/L	50	120	20	50	120	20		
Carbon tetrachloride	56-23-5	1	0.5	0.26	ug/L	70	125	20	70	125	20		
Chlorobenzene	108-90-7	1	0.5	0.14	ug/L	70	120	20	70	120	20		
Chloroethane	75-00-3	1	0.5	0.34	ug/L	50	150	20	50	150	20		
Chloroform	67-66-3	1	0.5	0.2	ug/L	70	120	20	70	120	20		
Chloromethane	74-87-3	1	0.5	0.18	ug/L	50	134	20	50	134	20		
2-Chloroethyl vinyl ether	110-75-8	2	1	0.78	ug/L	40	130	20	40	130	20		
1,2-Dibromo-3-Chloropropane	96-12-8	2	1.2	0.87	ug/L	60	121	20	60	121	20		
Dibromochloromethane	124-48-1	1	0.5	0.32	ug/L	70	120	20	70	120	20		
Dibromomethane	74-95-3	1	0.5	0.33	ug/L	70	120	20	70	120	20		
Dichlorodifluoromethane	75-71-8	1	0.5	0.2	ug/L	40	140	20	40	140	20		
1,2-Dibromoethane	106-93-4	1	0.5	0.36	ug/L	70	120	20	70	120	20		
Ethylbenzene	100-41-4	0.5	0.25	0.13	ug/L	75	120	20	75	120	20		
Hexachlorobutadiene	87-68-3	1	0.5	0.26	ug/L	70	135	20	70	135	20		
Isopropylbenzene	98-82-8	1	0.5	0.14	ug/L	70	120	20	70	120	20		
p-Isopropyltoluene	99-87-6	1	0.5	0.17	ug/L	70	120	20	70	120	20		
Methyl Ethyl Ketone	78-93-3	5	2.5	1.47	ug/L	54	138	20	54	138	20		
1-Chlorohexane	544-10-5	1	0.5	0.36	ug/L	70	120	20	70	120	20		
Methylene Chloride	75-09-2	5	2.5	0.68	ug/L	65	125	20	65	125	20		
m&p-Xylene	179601-23-1	1	0.5	0.26	ug/L	75	120	20	75	120	20		
Methyl tert-butyl ether	1634-04-4	1	0.5	0.24	ug/L	58	122	20	58	122	20		
Naphthalene	91-20-3	1	0.5	0.16	ug/L	55	132	20	55	132	20		
2-Chlorotoluene	95-49-8	1	0.5	0.21	ug/L	70	120	20	70	120	20		
o-Xylene	95-47-6	0.5	0.25	0.068	ug/L	70	120	20	70	120	20		
Tetrachloroethene	127-18-4	1	0.5	0.17	ug/L	70	123	20	70	123	20		
4-Chlorotoluene	106-43-4	1	0.5	0.2	ug/L	70	120	20	70	120	20		
N-Propylbenzene	103-65-1	1	0.5	0.13	ug/L	70	120	20	70	120	20		
sec-Butylbenzene	135-98-8	1	0.5	0.15	ug/L	70	120	20	70	120	20		
Styrene	100-42-5	1	0.5	0.1	ug/L	75	120	20	75	120	20		
trans-1,2-Dichloroethene	156-60-5	1	0.5	0.25	ug/L	70	124	20	70	124	20		
trans-1,3-Dichloropropene	10061-02-6	1	0.5	0.21	ug/L	70	120	20	70	120	20		
tert-Butylbenzene	98-06-6	1	0.5	0.14	ug/L	70	120	20	70	120	20		

(070-006)

Trichloroethene	79-01-6	0.5	0.25	0.19	ug/L	70	120	20	70	120	20		
Trichlorofluoromethane	75-69-4	1	0.5	0.19	ug/L	63	134	20	63	134	20		
Toluene	108-88-3	0.5	0.25	0.11	ug/L	70	120	20	70	120	20		
Vinyl chloride	75-01-4	0.5	0.25	0.1	ug/L	62	138	20	62	138	20		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/L							75	125
Toluene-d8 (Surr)	2037-26-5				ug/L							75	120
4-Bromofluorobenzene (Surr)	460-00-4				ug/L							75	120
Dibromofluoromethane	1868-53-7				ug/L							75	120
Dichlorofluoromethane	75-43-4	1	0.5	0.33	ug/L	50	120	20	50	120	20		

MDL W GCMS VOA	Purge and Trap	5030B

(040007)

MDL W GCMS VOA APIX	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
3-Chloropropene	107-05-1	2.5	1.2	0.48	ug/L								
2-Chloro-1,3-butadiene	126-99-8	1	0.5	0.15	ug/L								
Ethyl methacrylate	97-63-2	2.5	1.2	0.73	ug/L								
Iodomethane	74-88-4	2.5	1.2	0.53	ug/L	49	140	20	49	140	20		
Isobutanol	78-83-1	100	50	25.3	ug/L								
Methacrylonitrile	126-98-7	2.5	1.2	0.7	ug/L								
Methyl methacrylate	80-62-6	2.5	1.2	0.79	ug/L								
Pentachloroethane	76-01-7	10	1.2	0.63	ug/L								
trans-1,4-Dichloro-2-butene	110-57-6	10	1.2	1.81	ug/L								
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/L							75	125
Toluene-d8 (Surr)	2037-26-5				ug/L							75	120
4-Bromofluorobenzene (Surr)	460-00-4				ug/L							75	120
Dibromofluoromethane	1868-53-7				ug/L							75	120
1,3,5-Trichlorobenzene	108-70-3	1	0.5	0.17	ug/L	70	121	20	70	121	20		
Ethyl ether	60-29-7	1	0.5	0.31	ug/L	55	120	20	55	120	20		
Hexane	110-54-3	1	0.5	0.13	ug/L	50	120	20	50	120	20		
Isopropyl ether	108-20-3	1	0.5	0.15	ug/L								
Ethyl acetate	141-78-6	10	5	3.17	ug/L								
Heptane	142-82-5	1	0.5	0.12	ug/L	60	127	20	60	127	20		
2-Methylnaphthalene	91-57-6	1	0.5	0.18	ug/L								
Butadiene	106-99-0	1	0.5	0.21	ug/L								
n-Butyl alcohol	71-36-3	100	50	49.6	ug/L								
2-Nitropropane	79-46-9	100	50	18.4	ug/L								
Cyclohexanone	108-94-1	100	50	45	ug/L								
Cyclohexane	110-82-7	1	0.5	0.23	ug/L	51	134	20	51	134	20		
Methylcyclohexane	108-87-2	1	0.5	0.14	ug/L	59	120	20	59	120	20		
Methyl acetate	79-20-9	2	1	0.53	ug/L	50	130	20	50	130	20		
2,2,4-Trimethylpentane	540-84-1	1	0.5	0.5	ug/L								

MDL W GCMS VOA	Purge and Trap	5030B

(040-008)

MDL S MSVOA Default List	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
1,1,1,2-Tetrachloroethane	630-20-6	2	1	0.346	ug/Kg	75	120	30	75	120	30		
Chloromethane	74-87-3	2	1	0.462	ug/Kg	50	134	30	50	134	30		
Vinyl chloride	75-01-4	0.25	0.12	0.104	ug/Kg	62	138	30	62	138	30		
Bromomethane	74-83-9	2	1	0.682	ug/Kg	50	150	30	50	150	30		
Chloroethane	75-00-3	2	1	0.435	ug/Kg	50	150	30	50	150	30		
Trichlorofluoromethane	75-69-4	2	1	0.415	ug/Kg	63	134	30	63	134	30		
Acrolein	107-02-8	40	20	8.27	ug/Kg	40	130		40	130			
1,1-Dichloroethene	75-35-4	1	0.5	0.307	ug/Kg	58	122	30	58	122	30		
Carbon disulfide	75-15-0	5	2.5	0.427	ug/Kg	50	120	30	50	120	30		
Acetone	67-64-1	5	2.5	1.3	ug/Kg	46	153	30	46	153	30		
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	2	1	0.382	ug/Kg	46	120	30	46	120	30		
Methylene Chloride	75-09-2	5	2.5	0.683	ug/Kg	65	125	30	65	125	30		
trans-1,2-Dichloroethene	156-60-5	1	0.5	0.25	ug/Kg	70	124	30	70	124	30		
Methyl tert-butyl ether	1634-04-4	2	1	0.43	ug/Kg	58	122	30	58	122	30		
Acetonitrile	75-05-8	16	8	7.28	ug/Kg	60	131		60	131			
1,1-Dichloroethane	75-34-3	1	0.5	0.185	ug/Kg	68	121	30	68	121	30		
2,2-Dichloropropane	594-20-7	1	0.5	0.316	ug/Kg	67	125	30	67	125	30		
cis-1,2-Dichloroethene	156-59-2	1	0.5	0.123	ug/Kg	70	120	30	70	120	30		
Methyl Ethyl Ketone	78-93-3	5	2.5	1.47	ug/Kg	54	138	30	54	138	30		
Acrylonitrile	107-13-1	8	4	2.08	ug/Kg	62	127		62	127			
Bromochloromethane	74-97-5	2	1	0.378	ug/Kg	67	122	30	67	122	30		
Chloroform	67-66-3	1	0.5	0.205	ug/Kg	70	120	30	70	120	30		
Vinyl acetate	108-05-4	2	1	0.334	ug/Kg	50	130	30	50	130	30		
1,1,1-Trichloroethane	71-55-6	1	0.5	0.201	ug/Kg	70	123	30	70	123	30		
1,1-Dichloropropene	563-58-6	1	0.5	0.344	ug/Kg	70	120	30	70	120	30		
Carbon tetrachloride	56-23-5	1	0.5	0.257	ug/Kg	70	125	30	70	125	30		
Benzene	71-43-2	0.25	0.12	0.0742	ug/Kg	70	120	30	70	120	30		
1,2-Dichloroethane	107-06-2	1	0.5	0.285	ug/Kg	69	120	30	69	120	30		
Propionitrile	107-12-0	16	8	3.5	ug/Kg	66	129	30	66	129	30		
Trichloroethene	79-01-6	0.5	0.25	0.186	ug/Kg	70	120	30	70	120	30		
Tetrahydrofuran	109-99-9	4	2	1.25	ug/Kg	50	133	30	50	133	30		
1,2-Dichloropropane	78-87-5	1	0.5	0.196	ug/Kg	70	120	30	70	120	30		
Dibromomethane	74-95-3	2	1	0.48	ug/Kg	70	120	30	70	120	30		
Dichlorodifluoromethane	75-71-8	2	1	0.513	ug/Kg	40	140	30	40	140	30		

(040-004)

Bromodichloromethane	75-27-4	2	1	0.338	ug/Kg	70	120	30	70	120	30		
cis-1,3-Dichloropropene	10061-01-5	1	0.5	0.178	ug/Kg	70	120	30	70	120	30		
methyl isobutyl ketone	108-10-1	5	2.5	0.332	ug/Kg	59	135	30	59	135	30		
1,2-Dichloroethene, Total	540-59-0	2	1	0.259	ug/Kg	70	124	30	70	124	30		
Toluene	108-88-3	0.25	0.151	0.115	ug/Kg	70	120	30	70	120	30		
trans-1,3-Dichloropropene	10061-02-6	1	0.5	0.208	ug/Kg	70	120	30	70	120	30		
1,1,2-Trichloroethane	79-00-5	1	0.5	0.279	ug/Kg	69	120	30	69	120	30		
Tetrachloroethene	127-18-4	1	0.5	0.167	ug/Kg	70	123	30	70	123	30		
1,3-Dichloropropane	142-28-9	1	0.5	0.134	ug/Kg	70	120	30	70	120	30		
2-Hexanone	591-78-6	5	2.5	0.562	ug/Kg	55	144	30	55	144	30		
Dibromochloromethane	124-48-1	2	1	0.346	ug/Kg	70	120	30	70	120	30		
2-Chloroethyl vinyl ether	110-75-8	2	1	0.779	ug/Kg	40	130	30	40	130	30		
1,2-Dibromoethane	106-93-4	2	1	0.314	ug/Kg	70	120	30	70	120	30		
Chlorobenzene	108-90-7	1	0.5	0.143	ug/Kg	70	120	30	70	120	30		
Ethylbenzene	100-41-4	0.25	0.14	0.126	ug/Kg	75	120	30	75	120	30		
m&p-Xylene	179601-23-1	0.5	0.296	0.161	ug/Kg	75	120	30	75	120	30		
o-Xylene	95-47-6	0.25	0.129	0.0684	ug/Kg	70	120	30	70	120	30		
Styrene	100-42-5	1	0.5	0.0988	ug/Kg	75	120	30	75	120	30		
Bromoform	75-25-2	2	1	0.441	ug/Kg	70	125	30	70	125	30		
Isopropylbenzene	98-82-8	2	1	0.251	ug/Kg	70	120	30	70	120	30		
Bromobenzene	108-86-1	2	1	0.425	ug/Kg	70	120	30	70	120	30		
1,1,2,2-Tetrachloroethane	79-34-5	1	0.5	0.234	ug/Kg	70	128	30	70	128	30		
1,2,3-Trichloropropane	96-18-4	2	1	0.574	ug/Kg	70	120	30	70	120	30		
1-Chlorohexane	544-10-5	2	1	0.373	ug/Kg	70	120	30	70	120	30		
N-Propylbenzene	103-65-1	2	1	0.175	ug/Kg	70	120	30	70	120	30		
2-Chlorotoluene	95-49-8	1	0.5	0.207	ug/Kg	70	120	30	70	120	30		
1,3,5-Trimethylbenzene	108-67-8	2	1	0.206	ug/Kg	75	123	30	75	123	30		
4-Chlorotoluene	106-43-4	1	0.5	0.197	ug/Kg	70	120	30	70	120	30		
tert-Butylbenzene	98-06-6	1	0.5	0.136	ug/Kg	70	120	30	70	120	30		
1,2,4-Trimethylbenzene	95-63-6	2	1	0.211	ug/Kg	75	121	30	75	121	30		
sec-Butylbenzene	135-98-8	1	0.5	0.154	ug/Kg	70	120	30	70	120	30		
1,3-Dichlorobenzene	541-73-1	2	1	0.257	ug/Kg	70	120	30	70	120	30		
p-Isopropyltoluene	99-87-6	2	1	0.185	ug/Kg	70	120	30	70	120	30		
1,4-Dichlorobenzene	106-46-7	2	1	0.174	ug/Kg	75	120	30	75	120	30		
n-Butylbenzene	104-51-8	1	0.5	0.129	ug/Kg	75	120	30	75	120	30		
1,2-Dichlorobenzene	95-50-1	2	1	0.205	ug/Kg	75	120	30	75	120	30		
1,2-Dibromo-3-Chloropropane	96-12-8	2	1	0.871	ug/Kg	60	121	30	60	121	30		

(010-010)

1,2,4-Trichlorobenzene	120-82-1	2	1	0.378	ug/Kg	65	121	30	65	121	30		
Hexachlorobutadiene	87-68-3	2	1	0.346	ug/Kg	70	135	30	70	135	30		
Naphthalene	91-20-3	2	1	0.494	ug/Kg	55	132	30	55	132	30		
1,2,3-Trichlorobenzene	87-61-6	2	1	0.35	ug/Kg	56	137	30	56	137	30		
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							75	125
Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	120
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							75	120
Dibromofluoromethane	1868-53-7				ug/Kg							75	120
Dichlorofluoromethane	75-43-4	2	1	0.414	ug/Kg	50	120	30	50	120	30		

MDL S MSVOA Default List	Closed System Purge and Trap	5035A_M_Calc

(040-011)

MDL S MSVOA APIX	Volatile Organic Compounds (GC/MS)	8260B

Analyte Description	CAS Number	RL - Limit	LOD - Limit	MDL - Limit	Units	LCS Rec LCL	LCS Rec UCL	LCS RPD	MS Rec LCL	MS Rec UCL	MS RPD	Surr LCL	Surr UCL
3-Chloropropene	107-05-1	4	2	0.476	ug/Kg								
2-Chloro-1,3-butadiene	126-99-8	2	1	0.152	ug/Kg								
Iodomethane	74-88-4	4	2	0.528	ug/Kg	49	140	30	49	140	30		
Isobutanol	78-83-1	200	100	25.3	ug/Kg								
Methacrylonitrile	126-98-7	4	2	0.7	ug/Kg								
Methyl methacrylate	80-62-6	4	2	0.79	ug/Kg								
Pentachloroethane	76-01-7	16	2	0.626	ug/Kg								
trans-1,4-Dichloro-2-butene	110-57-6	16	2	1.81	ug/Kg								
1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							75	125
Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	120
4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							75	120
Dibromofluoromethane	1868-53-7				ug/Kg							75	120
1,3,5-Trichlorobenzene	108-70-3	2	1	0.167	ug/Kg	70	121	30	70	121	30		
Ethyl ether	60-29-7	2	1	0.315	ug/Kg	55	120	30	55	120	30		
Hexane	110-54-3	2	1	0.126	ug/Kg	50	120	30	50	120	30		
Isopropyl ether	108-20-3	2	1	0.147	ug/Kg								
Ethyl acetate	141-78-6	20	10	3.17	ug/Kg								
Heptane	142-82-5	2	1	0.124	ug/Kg	60	127	30	60	127	30		
2-Methylnaphthalene	91-57-6	2	1	0.183	ug/Kg								
Butadiene	106-99-0	2	1	0.208	ug/Kg								
n-Butyl alcohol	71-36-3	200	100	49.6	ug/Kg								
2-Nitropropane	79-46-9	200	100	18.4	ug/Kg								
Cyclohexanone	108-94-1	200	100	45	ug/Kg								
Cyclohexane	110-82-7	2	1	0.23	ug/Kg	51	134	30	51	134	30		
Methylcyclohexane	108-87-2	2	1	0.14	ug/Kg	59	120	30	59	120	30		
Methyl acetate	79-20-9	2	1	0.526	ug/Kg	50	130	30	50	130	30		
2,2,4-Trimethylpentane	540-84-1	2	1	1	ug/Kg								

MDL S MSVOA APIX	Closed System Purge and Trap	5035A_M_Calc

(040-012)

Table 2

Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Benzene	78	
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon tetrachloride	117	119
Chlorobenzene	112	77,114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110,77
Ethylbenzene	91	106
Hexachlorobutadiene	225	223,227
Isopropylbenzene	105	120
p-Isopropylbenzene	119	134, 91
Methylene chloride	84	86,49
Naphthalene	128	86, 49
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129,131,166

Table 2
(continued)
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
Internal Std./Surrogates		
4-Bromofluorobenzene (S)	95	174, 176
1,4-Dichlorobenzene-d_4 (IS)	152	115, 150
Pentafluorobenzene (IS)	168	
Chlorobenzene-d_5 (IS)	117	
1,4-Difluorobenzene (IS)	114	
1,2-Dichloroethane-d_4 (S)	65	
Toluene-d_8 (S)	98	
Dibromofluoromethane (S)	113	

***NOTE:** The primary and secondary ions listed here are taken directly from SW-846 Method 8260. The laboratory uses secondary ions in the cases of Ethylbenzene, Toluene, 1,1,2-Trichloroethane, Trichloroethene, 1,2,3-Trichloropropane and Xylenes due to interferences and/or to maintain consistency between methods.

Figure 1.

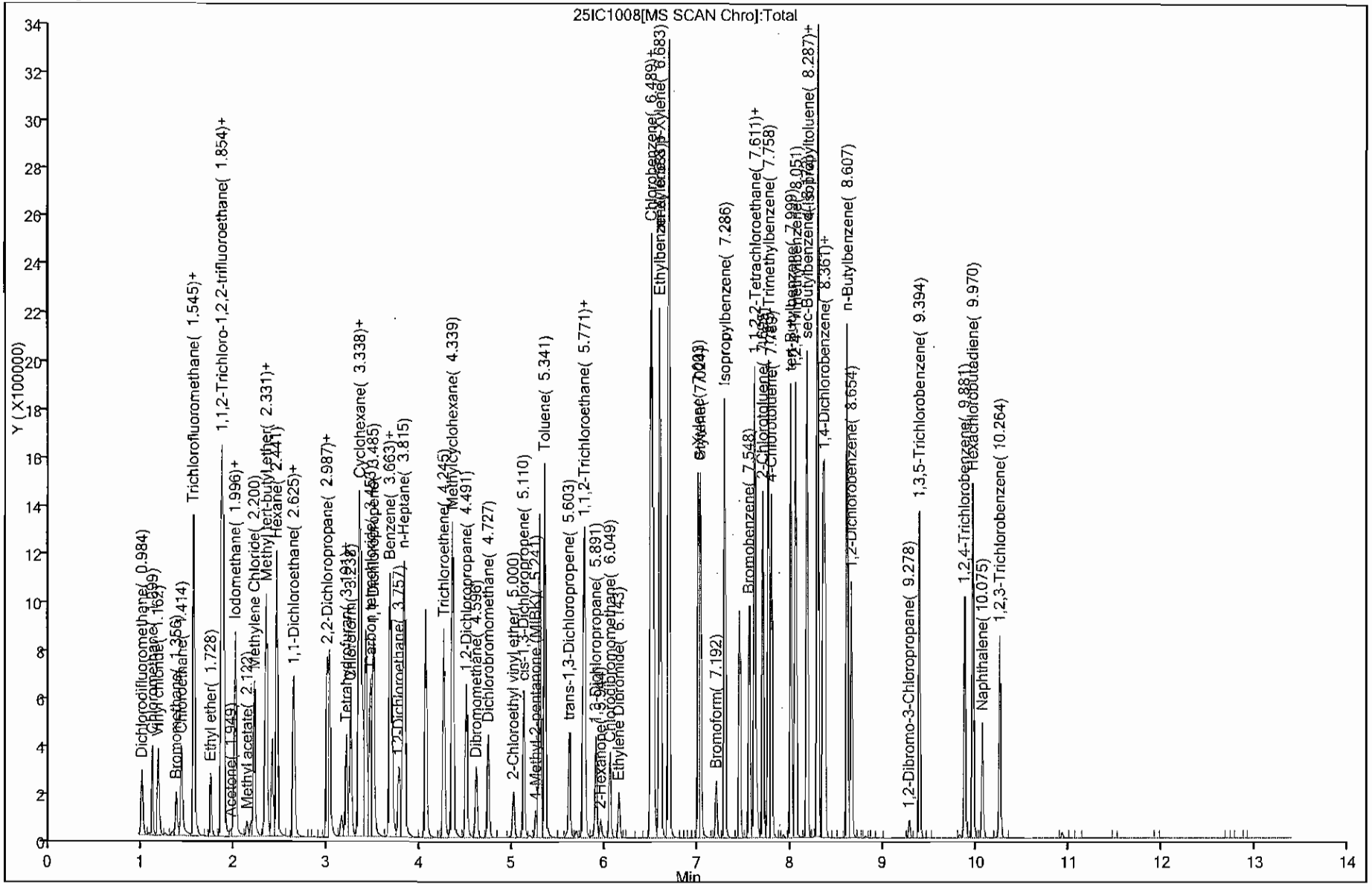
Example: Total Ion Chromatogram for 5 mL Purge Water (043-001)

Report Date: 05-Dec-2012 16:17:37 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago

Data File:	\\Chi-svr07\ChromData\CMS25\20121008-13136.b\25IC1008.D		
Injection Date:	09-Oct-2012 03:52:30	Limit Group:	MSVOA_8260_ICAL_WATER
Client ID:		Instrument ID:	CMS25
Lims Batch ID:	13136	Lims Sample ID:	11
Operator ID:	BDA	Purge Vol:	5.000 mL
Column Type:		Column Dia:	
Y Scaling:			



(100-ch0)

Figure 2.

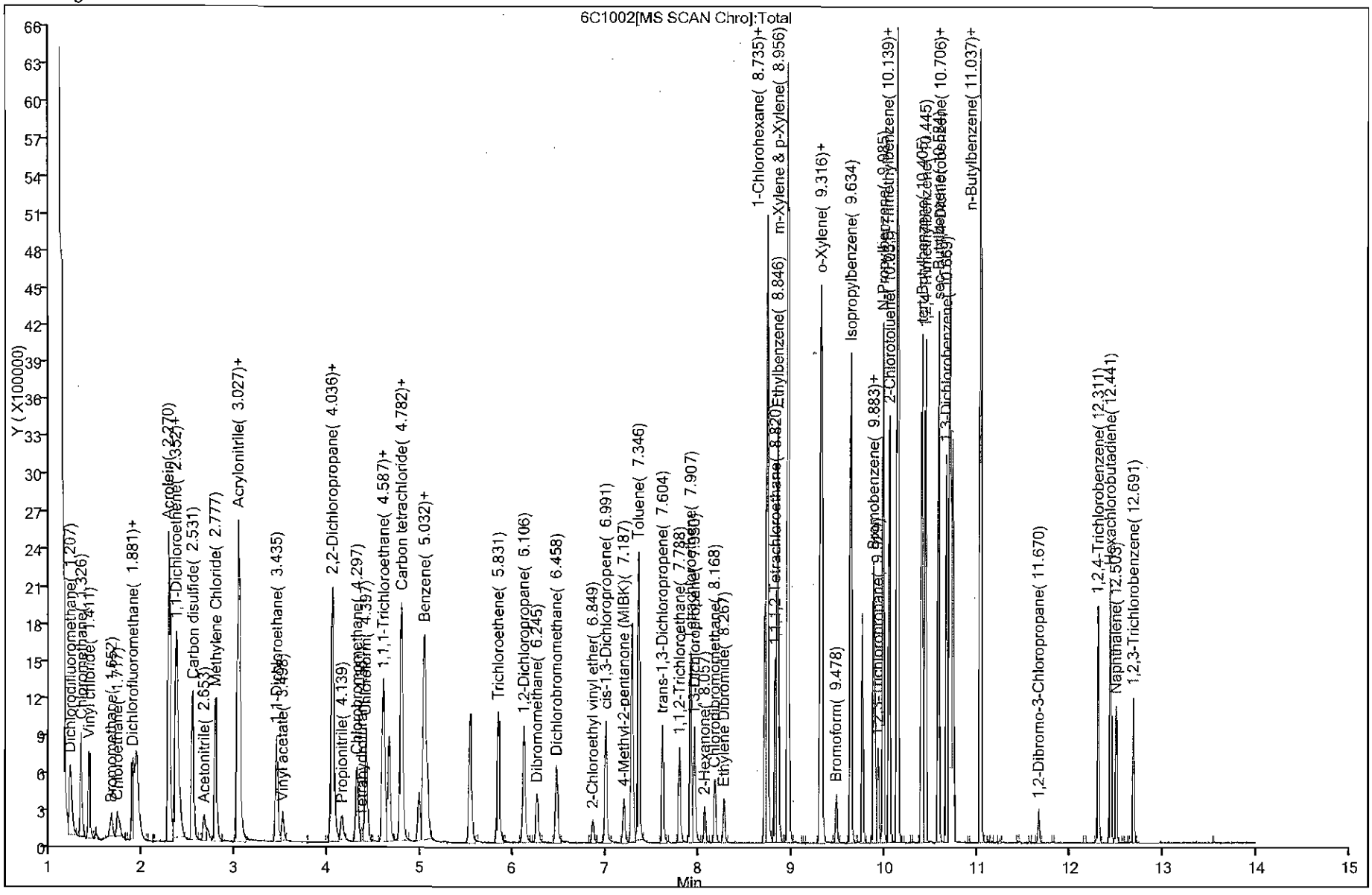
Example: Total Ion Chromatogram for 5 mL Purge Soil (044-001)

Report Date: 05-Dec-2012 16:18:40 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago

Data File:	\\chi-svr07\chromdata\CMS06\20121002-13033.b\6C1002.d		
Injection Date:	02-Oct-2012 14:42:30	Limit Group:	MSVOA_8260_ICAL_SOIL_LOW
Client ID:		Instrument ID:	CMS06
Lims Batch ID:	13033	Lims Sample ID:	5
Operator ID:	WBS	Purge Vol:	5.000 mL
Column Type:		Column Dia:	
Y Scaling:			



(100-440)

Attachment 1.

Example: Initial Calibration Guides, Method Listings, Concentrator Conditions, Flow Settings (045-001 to 045-004)

8260 5ml WATER/ HIGH LEVEL SOIL ICAL 1/ 624

TUNE FILE: _____ MATRIX: WATER AND HIGH LEVEL SOIL (NOT HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

LEVEL CHROM WATER	CONC WATER	FILE NAME	AMOUNT INJECTED	ADD MEOH
1 IC	0.25ppb	__I____A	5ul IS + 2.5ul LOW LOW 8260 STD	92.5ul
2 IC	1ppb	__I____B	5ul IS + 1ul LOW 8260 STD + 0.5ul LOW ACRL/NIT STD	93.5ul
3 IC	2ppb	__I____C	5ul IS + 2ul | + 1ul |	92ul
4 IC	5ppb	__I____D	5ul IS + 5ul | + 2.5ul |	87.5ul
5 IC	20ppb	__I____E	5ul IS + 2ul SS + 20ul ⊥ + 10ul ⊥	65ul
6 ICIS	50ppb	__I____F	GAS STD, 8260 STD, KET/CS2 STD 5ul IS + 5ul SS + 2.5ul @ VA/CEVE STD, ACRL/NIT STD, DCFM STD	75ul
7 IC	100ppb	__I____G	5ul IS + 10ul SS+ 5ul |	55ul
8 IC	150ppb	__I____H	5ul IS + 15ul SS+ 7.5ul |	35ul
9 IC	200ppb	__I____I	5ul IS + 20ul SS+ 10ul ⊥	15ul

ICV 50ppb	__S_____	5ul IS/SS + 5ul @GAS/DCFM SPK, VOC SPK, BCM++SPK, CYCLOHEX++SPK	80ul

STD NAME	CONC
8260 LOW IS	50ppm
8260 LOWSS	50ppm
GAS STD WK	100ppm
8260 STD WK	100ppm
KET / CS2 STDWK	100ppm
VA / 2CEVE STDWK	100ppm
ACROLEIN/NITRILES STDWK	4000/800ppm
DICHLOROFLOROMETHANE STD	100ppm
LOW 8260 STD WK	5ppm
LOW LOW 8260 STD WK	0.5ppm
LOW ACROLEIN/NITRILES STD WK	400/80ppm

CHI-22-20-059/D-12/12

(045-001)

8260 5ML WATER/ HIGH LEVEL SOIL ICAL2

TUNE FILE: _____ MATRIX: WATER AND HIGH LEVEL SOIL (NOT HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

LEVEL CHROM	CONC WATER ICAL2	CONC WATER APIX	FILE NAME	AMOUNT INJECTED	ADD MEOH
2 IC	1ppb	2.5ppb	__J____A	5ul IS + 1ul LOW ICAL2 + 0.5ul LOW APIX	93.5ul
3 IC	2ppb	5ppb	__J____B	5ul IS + 2ul LOW ICAL2 + 1ul LOW APIX	92ul
4 IC	5ppb	10ppb	__J____C	5ul IS + 5ul | + 2ul |	88ul
5 IC	20ppb	40ppb	__J____D	5ul IS + 20ul ⊥ + 8ul ⊥	67ul
6 IC	50ppb	100ppb	__J____E	5ul IS + 2.5ul ICAL2 / 224TMP / CLPRENE + 2ul APIX	90.5ul
7 IC	100ppb	200ppb	__J____F	5ul IS + 5ul | + 4ul |	86ul
8 IC	150ppb	300ppb	__J____G	5ul IS + 7.5ul | + 6ul |	81.5ul
9 IC	200ppb	400ppb	__J____H	5ul IS + 10ul ⊥ + 8ul ⊥	77ul

STD NAME	CONC
8260 LOW IS	50ppm
ICAL2 STD	100ppm
2,2,4 TRIMETHYLPENTANE	100ppm
CLHOROPRENE	100ppm
APIX STD	250/10000ppm
LOW ICAL2	5ppm
LOW APIX	25/1000ppm

CHI-22-20-060/D-12/12

(045-002)

8260 SOIL - ICAL 1

TUNE FILE: _____ MATRIX: SOIL (HEATED)

TUNE TIME: _____ **INSTRUMENT NO:** _____

IS ARCHON INJECTION

STD LEVELS	CONC SOIL	FILE NAME	AMOUNT INJECTED
1 IC	5ppb	__I____A	ARCH 1ul IS + 5ul LOW 8260 STD + 2.5ul LOW ARC/NIT STD
2 IC	20ppb	__I____B	| + 2ul 8260 LOWSS + 20ul ⊥ + 10ul ⊥
3 ICIS	50ppb	__I____C	| + 5ul | + 2.5ul @GAS, 8260, KET+CS2 ,VA+CEVE ,ACR/NIT STD
4 IC	100ppb	__I____D	| + 10ul | + 5ul |
5 IC	150ppb	__I____E	| + 15ul | + 7.5ul |
6 IC	200ppb	__I____F	⊥ + 20ul ⊥ + 10ul ⊥

ICV	50ppb	__S____	1ul IS/SS (ARCHON) + 5ul @ GAS SPK, VOC SPK, BDCM++SPK, 5ul MEAC++SPK + 2.5ul ACR/NIT STD

STD NAME	CONC
8260 IS	250ppm
8260 LOW SS	50ppm
GAS STD	100ppm
8260 STD	100ppm
KET + CS2	100ppm
VA + 2-CEVE	100ppm
ARC/NITS STD WK	4000ppm/ 800ppm
LOW 8260 STD	5ppm
LOW ACR/NITR	400ppm/ 80ppm

CHI-22-20-061/D-12/12

(045-003)

8260 SOIL - ICAL 2

TUNE FILE: _____ MATRIX: SOIL (HEATED)

TUNE TIME: _____ INSTRUMENT NO: _____

STD LEVELS	CONC SOIL		FILE NAME	AMOUNT INJECTED
	ICAL2	APIX		
1 IC	5ppb	10ppb	__J____A	ARCH 1ul IS + 5ul LOW ICAL2 + 2ul LOW APIX
2 IC	20ppb	40ppb	__J____B	1ul IS + 20ul ⊥ + 8ul ⊥
3 IC	50ppb	100ppb	__J____C	1ul IS + 2.5ul ICAL2 / 224TMP / CLPRENE + 2ul APIX
4 IC	100ppb	200ppb	__J____D	1ul IS + 5ul | + 4ul APIX
5 IC	150ppb	300ppb	__J____E	1ul IS + 7.5ul | + 6ul |
6 IC	200ppb	400ppb	__J____F	1ul IS + 10ul ⊥ + 8ul ⊥

STD NAME	CONC
8260 IS	250ppm (ARCHON INJ)
ICAL2 STD	100ppm
2,2,4 TRIMETHYLPENTANE	100ppm
CLHOROPRENE	100ppm
APIX STD	250/10000ppm
LOW ICAL2	5ppm
LOW APIX	25/1000ppm

CHI-22-20-083/B-12/12

(045-004)

Example: Volatiles Method for 5972

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 21.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Example: Volatiles Method for 5973

GC Oven Parameters

Initial Temperature = 50 °C
Initial Time = 2.0 minutes
Aux Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
15.0	220	0.00

Run Time = 13.33 min.

Inlet Pressure Program

Mode=split
Gas = Helium
Column length = 25 m
Column Diameter = 0.25 mm
Constant flow = 1.0 mL/min
Injection port temp. = 250 °C
Program Time = 7.0 min.
Split ratio = 80:1
Gas saver = off

Scan Parameters

Mass Range = 35-260
Threshold = 100
Scans/sec = 6
EM Voltage = 1938
Solvent Delay = 0.8 min. (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Concentrator Conditions

Trap Temp. Prior to Purge	< 35
Desorb Preheat	250
Desorb	250
Bake	260
Purge Time	11 min
Desorb	0.5 - 2 min (inst. dependent)
Bake Time	4 min

Trap = Vocarb 3000

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	~40 mLs/min

Flow Adjustment

Capillary Column: 5972/MSD's;

· Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.

· Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is ≤ 1 mL/minute)

Approximate Vacuums

~5 x 10^{-6} torr

Example Archon Conditions

Transfer Line Temp	110 deg C
Soil Valve	95 deg C
Purge Pressure	25 psi
Purge Flow	~ 40 ml/min
Purge Time	11 min
Desorb	0.5 -2 min
SamplePre-heat(soils)	40 deg C ~ 2 min

Attachment 2.

Example: Target and Internal Standards; Initial Calibration (Form 6) (049-001 to 049-006)

TestAmerica Laboratories
Initial Calibration Report

Method: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\8260W25c.m
Instrument: CMS25 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 16-Oct-2012 08:26:47
No.Compounds:118

Initial Calibration Batches

Ical Batch: \\Chi-svr07\ChromData\CMS25\20120926-12901.b
Inj Date : 26-Sep-2012 08:40:30, Sublist: chrom-8260W25c*sub3

Ical Batch: \\Chi-svr07\ChromData\CMS25\20121009-13144.b
Inj Date : 09-Oct-2012 08:51:30, Sublist: chrom-8260W25c*sub4

Limit Group: MSVOA_8260_ICAL_WATER

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
1 Dichlorodifluoromethan		0.5670079	0.5177011	0.5059069	0.4690858	0.4443685	0.4472840	0.4434343	0.4507967		0.4806982		Avg 9.4
2 Chloromethane		0.7983395	0.6714810	0.6390374	0.5951559	0.5840725	0.5745356	0.5507310	0.5722328		0.6231982		Avg 13
3 Vinyl chloride	0.6381654	0.6964479	0.6376347	0.6017541	0.5811343	0.5921063	0.5899551	0.5821860	0.6010926		0.6133863		Avg 6.2
4 Butadiene		0.6346372	0.5522886	0.5385983	0.5128470	0.6471360	0.6356398	0.6194411	0.6435599		0.5980185		Avg 9.1
5 Bromomethane		0.2317090	0.2145561	0.1963735	0.1947667	0.2428464	0.2643693	0.2609119	0.2702086		0.2344677		Avg 13
6 Chloroethane		0.5053069	0.4984725	0.5276181	0.4914745	0.4963092	0.4845056	0.4691379	0.4791649		0.4939987		Avg 3.6
7 Trichlorofluoromethane		0.9923110	0.8871162	0.8971192	0.8574702	0.8841981	0.8660215	0.8515805	0.8811903		0.8896259		Avg 5.0
8 Dichlorofluoromethane		1.1350535	1.0293688	1.0323459	0.9635163	0.9870664	0.9652913	0.9520890	0.9780832		1.0053518		Avg 6.0
9 Ethyl ether		0.2922381	0.2281496	0.2200460	0.1988271	0.2343308	0.2225749	0.2212917	0.2166482		0.2292633		Avg 12
10 1,1,2-Trichloro-1,2,2-		0.5753102	0.5077434	0.4850567	0.4631406	0.5013407	0.4918017	0.4834973	0.4877003		0.4994489		Avg 6.7
11 Acrolein		0.0452026	0.0380158	0.0353227	0.0331448	0.0352003	0.0341237	0.0333766	0.0350008		0.0361734		Avg 11
12 1,1-Dichloroethene		0.4216237	0.4015936	0.3809182	0.3701530	0.4006299	0.3939610	0.3883340	0.3916693		0.3936103		Avg 3.9
13 Acetone				0.0743045	0.0505701	0.0466142	0.0438930	0.0405895	0.0436045	0.1622128	0.0420440		Linr 0.999
14 Iodomethane		0.3093932	0.3042478	0.3510302	0.4442501	0.5758859	0.5840020	0.5879890	0.5974957	-1.2925101	0.5897864		Linr 0.998
15 Carbon disulfide				1.2946763	1.2398188	1.3829146	1.3661924	1.3283942	1.3211056		1.3221836		Avg 3.9
16 3-Chloro-1-propene		0.2559416	0.2062609	0.1978888	0.2038497	0.2319763	0.2231576	0.2199005	0.2229339		0.2202387		Avg 8.4
17 Methyl acetate		0.1969322	0.1548250	0.1319946	0.1233530	0.1457672	0.1333607	0.1361298	0.1346567	0.0431239	0.1349694		Linr 0.999
18 Acetonitrile		0.0335157	0.0279661	0.0200524	0.0200103	0.0197977	0.0191075	0.0187218	0.0196007	0.1146493	0.0191369		Linr 0.999
19 Methylene Chloride				0.3665252	0.3366462	0.3545308	0.3456918	0.3423092	0.3521244		0.3496379		Avg 3.0
20 Methyl tert-butyl ethe		0.5465352	0.5193689	0.4930861	0.4686186	0.5014517	0.5032177	0.4991697	0.5307477		0.5077744		Avg 4.7
21 trans-1,2-Dichloroethe		0.4859663	0.4225881	0.4251265	0.4024584	0.4368672	0.4249428	0.4272579	0.4316629		0.4321088		Avg 5.5
22 Acrylonitrile		0.0807467	0.0664724	0.0627751	0.0606077	0.0611782	0.0604687	0.0588729	0.0615144		0.0640795		Avg 11
23 Hexane		0.8697920	0.7612921	0.7296100	0.7108560	0.8665634	0.8534431	0.8322823	0.8407833		0.8080778		Avg 7.9
24 Isopropyl ether		0.9740266	0.8990408	0.9385414	0.9234477	1.1264906	1.1087485	1.1028767	1.0850031		1.0197719		Avg 9.3
25 1,1-Dichloroethane		0.8591915	0.8057870	0.8223280	0.7846353	0.8351192	0.8202836	0.8169488	0.8298871		0.8217726		Avg 2.6

(100-640)

Method: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\8260W25c.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
26 Vinyl acetate		0.3014293	0.2454592	0.2511005	0.2378230	0.2502697	0.2573763	0.2582848	0.2703584		0.2590127		Avg 7.6
27 2-Chloro-1,3-butadiene		0.7394737	0.6994035	0.7017099	0.7042303	0.8620337	0.8402589	0.8242944	0.8316313		0.7753794		Avg 9.1
28 2,2-Dichloropropane		0.6031417	0.5430123	0.5567222	0.5571294	0.6053694	0.6090981	0.6089236	0.6198641		0.5879076		Avg 5.1
29 cis-1,2-Dichloroethene		0.4740790	0.4093930	0.4181827	0.4036312	0.4334885	0.4306231	0.4279273	0.4390061		0.4295414		Avg 5.1
30 2-Butanone (MEK)				0.0681093	0.0530397	0.0553904	0.0595293	0.0572424	0.0603843		0.0589492		Avg 8.9
31 Ethyl acetate		0.1494076	0.1213555	0.1205686	0.1218434	0.1409011	0.1334411	0.1341783	0.1307910		0.1315608		Avg 7.8
32 Propionitrile		0.0195367	0.0162800	0.0164229	0.0155197	0.0162471	0.0161478	0.0159251	0.0163593		0.0165548		Avg 7.5
34 Tetrahydrofuran				0.0426549	0.0368680	0.0363829	0.0381202	0.0383146	0.0405240		0.0388108		Avg 6.1
33 Chlorobromomethane		0.1721779	0.1579494	0.1690848	0.1543945	0.1677166	0.1670123	0.1650764	0.1709714		0.1655479		Avg 3.8
35 Methacrylonitrile		0.1026495	0.0911770	0.0846593	0.0839595	0.0985408	0.0941164	0.0933015	0.0922026		0.0925758		Avg 6.8
36 Chloroform		0.7323930	0.6730507	0.6728955	0.6331300	0.6709755	0.6626555	0.6556195	0.6667097		0.6709287		Avg 4.2
37 Cyclohexane		0.8926655	0.8034136	0.7869775	0.7889519	0.9708235	0.9564810	0.9343017	0.9447735		0.8847985		Avg 9.0
38 1,1,1-Trichloroethane		0.7093731	0.6492111	0.6583154	0.6306890	0.6846688	0.6813376	0.6714224	0.6848482		0.6712332		Avg 3.7
$ 39 Dibromofluoromethane					0.3780815	0.3628491	0.3606858	0.3492881	0.3611235		0.3624056		Avg 2.8
* 40 Pentafluorobenzene	536538	529975	509657	534288	524371	576562	567494	592550	587313				
41 Carbon tetrachloride		0.5738646	0.5486287	0.4874022	0.5056941	0.5451427	0.5350807	0.5306961	0.5244136		0.5313653		Avg 5.0
42 1,1-Dichloropropene		0.7442804	0.6383705	0.5967755	0.5810485	0.6196818	0.6099457	0.6065142	0.6105914		0.6259010		Avg 8.1
43 Isobutyl alcohol		0.0064466	0.0055691	0.0060748	0.0061744	0.0075918	0.0072573	0.0074027	0.0073092		0.0067283		Avg 11
44 Isooctane		1.6521281	1.5006154	1.5184290	1.4747837	1.7916633	1.7360101	1.7069912	1.7004278		1.6351311		Avg 7.4
45 Benzene	1.2632381	1.2658083	1.1778359	1.1598635	1.1419923	1.2361480	1.2062370	1.2083888	1.1910653		1.2056197		Avg 3.6
$ 46 1,2-Dichloroethane-d4					0.2631414	0.2447427	0.2424465	0.2375018	0.2397557		0.2455176		Avg 4.2
47 1,2-Dichloroethane		0.3527346	0.3259735	0.3135800	0.2988685	0.3195080	0.3103728	0.3099310	0.3118618		0.3178538		Avg 5.1
48 n-Heptane		0.6212277	0.6134098	0.5400494	0.5579068	0.6817185	0.6564044	0.6578960	0.6563669		0.6231224		Avg 8.2
* 49 1,4-Difluorobenzene	698364	698684	669533	705721	687995	738016	735319	757360	772529				
51 n-Butanol		0.0014289	0.0014096	0.0013697	0.0017144	0.0022333	0.0021427	0.0022895	0.0022400	-0.1239109	0.0020714	0.0000000	Quad 0.998
50 Trichloroethene	0.3568340	0.4028287	0.3604005	0.3585836	0.3455003	0.3844442	0.3763237	0.3813316	0.3707527		0.3707777		Avg 4.7
52 Methylcyclohexane		0.5879476	0.5007386	0.5186824	0.5207446	0.6325503	0.6158819	0.6107887	0.6122142		0.5749435		Avg 9.2
53 1,2-Dichloropropane		0.3291187	0.2888207	0.2995235	0.2797404	0.3160826	0.3097268	0.3100397	0.3107958		0.3054810		Avg 5.1
54 Methyl methacrylate		0.1215269	0.1005078	0.0896640	0.0939875	0.1114429	0.1111653	0.1125914	0.1109657		0.1064814		Avg 10
55 Dibromomethane		0.1221582	0.1199717	0.1178511	0.1141069	0.1255013	0.1215976	0.1206762	0.1221223		0.1204982		Avg 2.8
56 Dichlorobromomethane		0.3657591	0.3278031	0.3381081	0.3197116	0.3479342	0.3424738	0.3443836	0.3462330		0.3415508		Avg 4.0
58 2-Nitropropane		0.0468651	0.0384237	0.0393263	0.0417088	0.0495180	0.0488121	0.0498640	0.0513239		0.0457302		Avg 11
57 2-Chloroethyl vinyl et			0.0746042	0.0794082	0.0800151	0.0944221	0.0960692	0.0966894	0.1000328		0.0887487		Avg 12
59 cis-1,3-Dichloropropen		0.3336988	0.3253387	0.3212884	0.3285598	0.3689839	0.3733958	0.3759058	0.3806226		0.3509742		Avg 7.4
60 4-Methyl-2-pentanone (				0.0875417	0.0815958	0.1022918	0.0969144	0.0941367	0.0996623		0.0936904		Avg 8.3
$ 61 Toluene-d8 (Surr)					1.1383876	1.1170110	1.1056990	1.1026817	1.0807983		1.1089155		Avg 1.9
62 Toluene	0.8359537	0.8078044	0.7141545	0.7102098	0.7439117	0.8019596	0.7896212	0.7958685	0.7825004		0.7757760		Avg 5.6
63 trans-1,3-Dichloroprop		0.2855368	0.2713832	0.2471515	0.2470694	0.2843570	0.2895152	0.2912384	0.2973380		0.2766987		Avg 7.1
64 Ethyl methacrylate		0.1760071	0.1397797	0.1510023	0.1605514	0.2016970	0.1991445	0.1975692	0.1994157		0.1781459		Avg 14
65 1,1,2-Trichloroethane		0.2116138	0.1930710	0.1986008	0.1860086	0.2036832	0.2041969	0.1983411	0.2092309		0.2005933		Avg 4.2
66 Tetrachloroethene		0.5375937	0.4771533	0.4740405	0.4617616	0.5268089	0.5121387	0.5158650	0.4937476		0.4998887		Avg 5.5
67 1,3-Dichloropropane		0.3275787	0.3165959	0.3148091	0.3068294	0.3303655	0.3242282	0.3188224	0.3222514		0.3201851		Avg 2.4
68 2-Hexanone				0.0654316	0.0630265	0.0767103	0.0727326	0.0715550	0.0728037		0.0703766		Avg 7.3
69 Chlorodibromomethane		0.2516396	0.2415007	0.2498342	0.2381211	0.2692521	0.2714422	0.2682245	0.2662930		0.2570384		Avg 5.2
70 Ethylene Dibromide		0.1512129	0.1306508	0.1431302	0.1346267	0.1536511	0.1525739	0.1514859	0.1545026		0.1464793		Avg 6.3

(04-002)

Method: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\8260W25c.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
71 1-Chlorohexane		0.7255059	0.5993737	0.5623933	0.5783930	0.6425900	0.6392658	0.6424442	0.6436176		0.6291980		Avg 8.0
* 72 Chlorobenzene-d5	583210	581387	564284	591152	579915	620764	618478	640528	662188				
73 Chlorobenzene		1.1088139	0.9782308	0.9717129	0.9416596	1.0187044	0.9909536	0.9856993	0.9572859		0.9941325		Avg 5.2
74 Ethylbenzene	0.6011557	0.6131888	0.5261092	0.5670792	0.5502013	0.6162084	0.6077751	0.6100165	0.5928320		0.5871740		Avg 5.5
75 1,1,1,2-Tetrachloroeth		0.3551851	0.3266972	0.3226074	0.3114250	0.3552123	0.3523335	0.3516167	0.3478944		0.3403714		Avg 5.1
76 m-Xylene & p-Xylene	1.4391043	1.4063782	1.2512095	1.2958342	1.2995999	1.4415253	1.4117103	1.4086722	1.3414010		1.3661594		Avg 5.2
77 o-Xylene	1.3216509	1.3507354	1.1703770	1.2144085	1.2208212	1.3886050	1.3682839	1.3637962	1.3310344		1.3033014		Avg 6.1
78 Styrene		0.9333714	0.8484646	0.9351740	0.9530578	1.0870878	1.0670008	1.0731823	1.0489480		0.9932858		Avg 8.8
80 Bromoform		0.1466321	0.1351713	0.1406576	0.1417190	0.1606230	0.1575828	0.1577870	0.1572227		0.1496744		Avg 6.5
81 Isopropylbenzene		3.5808953	3.2675645	3.4152701	3.4472224	3.8826406	3.8864490	3.8429598	3.7435616		3.6333204		Avg 6.6
82 Cyclohexanone		0.0237600	0.0187729	0.0171438	0.0180948	0.0258711	0.0244352	0.0216336	0.0226969		0.0215510		Avg 15
$ 83 4-Bromofluorobenzene (					0.4139270	0.4091668	0.4019634	0.4064334	0.3960665		0.4055114		Avg 1.7
84 Bromobenzene		0.8257190	0.7631013	0.7312698	0.7177809	0.8259765	0.8289167	0.8177552	0.8141248		0.7905805		Avg 5.8
85 1,1,2,2-Tetrachloroeth		0.3756010	0.3326013	0.3402062	0.3195597	0.3664511	0.3611532	0.3517063	0.3582313		0.3506888		Avg 5.3
86 N-Propylbenzene		4.4043078	3.7425144	4.0051334	4.0347777	4.5489634	4.4977686	4.4578255	4.3254803		4.2520964		Avg 6.8
87 1,2,3-Trichloropropane		0.3323102	0.2662447	0.2733496	0.2640240	0.2914115	0.2866047	0.2797277	0.2849616		0.2848292		Avg 7.5
88 trans-1,4-Dichloro-2-b		0.0455969	0.0428127	0.0425451	0.0466693	0.0585385	0.0578518	0.0561271	0.0572159		0.0509196		Avg 14
89 2-Chlorotoluene		2.3597041	2.1859513	2.1669850	2.1429723	2.3897555	2.3797157	2.3580117	2.3229944		2.2882612		Avg 4.6
90 1,3,5-Trimethylbenzene		2.6984014	2.4888285	2.6677287	2.7360948	3.0767749	3.0931746	3.0628816	3.0129485		2.8546041		Avg 8.2
91 4-Chlorotoluene		2.6144811	2.3010301	2.2918475	2.2543918	2.4884179	2.4684731	2.4905358	2.4303628		2.4174425		Avg 5.1
92 tert-Butylbenzene		2.4801732	2.3263910	2.4684525	2.5442826	2.8984751	2.8760048	2.8542360	2.8210050		2.6586275		Avg 8.5
93 Pentachloroethane		0.3183393	0.2491155	0.2380756	0.2374118	0.2834977	0.2669268	0.2602731	0.2647968		0.2648046		Avg 10
94 1,2,4-Trimethylbenzene		2.6966272	2.4062008	2.5889578	2.6586809	3.0077119	3.0010472	2.9939911	2.9188300		2.7840059		Avg 8.2
95 sec-Butylbenzene		4.0794493	3.6530693	3.8601866	3.8701054	4.3848201	4.3180957	4.2986561	4.1846106		4.0811241		Avg 6.4
96 1,3-Dichlorobenzene		1.8600855	1.6086485	1.5470634	1.5382912	1.7084484	1.7174716	1.7431273	1.7034955		1.6783289		Avg 6.4
97 4-Isopropyltoluene		3.1824069	2.9118747	3.1729660	3.2730876	3.7300333	3.7295078	3.7989827	3.6813691		3.4350285		Avg 9.8
* 98 1,4-Dichlorobenzene-d4	275295	281815	275029	293763	294585	314061	306532	320470	324227				
99 1,4-Dichlorobenzene		1.8966343	1.6252832	1.5911126	1.4849874	1.6369017	1.5997759	1.5974288	1.5632312		1.6244194		Avg 7.4
100 n-Butylbenzene		3.2063588	2.7853426	2.8989015	2.9148548	3.3384024	3.3077705	3.3720962	3.2003211		3.1280060		Avg 7.3
101 1,2-Dichlorobenzene		1.4647907	1.2295976	1.2910067	1.2452094	1.3866956	1.3616882	1.3396501	1.3331169		1.3314694		Avg 5.8
102 1,2-Dibromo-3-Chloropr			0.0346327	0.0489510	0.0421016	0.0528528	0.0498415	0.0487066	0.0505919		0.0468111		Avg 13
103 1,3,5-Trichlorobenzene		1.7897577	1.2495380	1.1661629	1.0925033	1.3431058	1.2646685	1.2547999	1.2166868	0.2516781	1.2407875		Linr 0.998
104 1,2,4-Trichlorobenzene		1.0155599	0.8304579	0.8525921	0.8065414	0.9639147	0.9555952	0.9841972	0.9507729		0.9199539		Avg 8.5
105 Hexachlorobutadiene		0.9783014	0.7522843	0.7143514	0.7056452	0.8384136	0.8170795	0.8387213	0.8158983		0.8075869		Avg 11
106 Naphthalene		1.0989479	0.8816343	0.9000453	0.9015649	1.1522762	1.1282199	1.1312583	1.1646917		1.0448298		Avg 12
107 1,2,3-Trichlorobenzene		0.8736228	0.7028350	0.6627792	0.6453910	0.7635555	0.7408721	0.7559699	0.7482065		0.7366540		Avg 9.6
108 2-Methylnaphthalene		0.8177208	0.4925193	0.3866647	0.3802149	0.5884923	0.6159081	0.6348888	0.6367513	-0.2215946	0.6187730		Linr 0.992

(049-003)

TestAmerica Laboratories
Initial Calibration Report

Method: \\chi-svr07\chromdata\CMS06\20121002-13033.b\8260S-06.m
Instrument: CMS06 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 03-Oct-2012 11:37:20
No.Compounds:118

Initial Calibration Batches

Ical Batch: \\chi-svr07\chromdata\CMS06\20121001-13011.b
Inj Date : 01-Oct-2012 17:56:30, Sublist: chrom-8260S-06*sub2

Limit Group: MSVOA_8260_ICAL_SOIL_LOW

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
1 Dichlorodifluoromethan	1.0899059	1.0342059	0.9105648	1.0312523	0.8991192	0.8485487		0.9689328		Avg 9.9
2 Chloromethane	1.3329093	1.3057975	1.0700024	1.2010168	1.0645766	1.0343664		1.1681115		Avg 11
3 Vinyl chloride	1.0082461	1.0047598	0.8270158	0.9360120	0.8300034	0.7934081		0.8999075		Avg 11
4 Butadiene	1.4732675	1.4220379	1.1686491	1.2165849	1.2187532	1.1729965		1.2787148		Avg 10
5 Bromomethane	0.2443729	0.2106417	0.1794482	0.1999972	0.1876646	0.1710083		0.1988555		Avg 13
6 Chloroethane	0.3972649	0.3434286	0.2760339	0.3475833	0.2917658	0.2728139		0.3214817		Avg 15
7 Dichlorofluoromethane	1.3451759	1.3110349	1.0345708	1.2200673	1.0612784	0.9717691		1.1573161		Avg 13
8 Trichlorofluoromethane	1.1121892	1.0179529	0.8621770	1.0439359	0.8963198	0.8310769		0.9606086		Avg 12
9 Ethyl ether	0.4264676	0.4969582	0.4083272	0.4457077	0.4434101	0.4224588		0.4405549		Avg 7.0
10 Acrolein	0.0865126	0.0816023	0.0791377	0.0755179	0.0720179	0.0692981		0.0773478		Avg 8.2
11 1,1-Dichloroethene	0.6280213	0.6137740	0.5098338	0.5793388	0.4834315	0.4844765		0.5498127		Avg 12
12 1,1,2-Trichloro-1,2,2-	0.8274168	0.7781534	0.6246866	0.7366379	0.6015244	0.6016783		0.6950162		Avg 14
13 Acetone	0.2113686	0.1898919	0.1664177	0.1651744	0.1488589	0.1585199		0.1733719		Avg 13
14 Iodomethane	0.9842623	0.8917911	0.9262624	0.8955757	0.8772760	0.8697159		0.9074806		Avg 4.7
15 Carbon disulfide	2.4385837	2.3468994	2.0477505	2.2610795	2.0015160	1.9339651		2.1716324		Avg 9.5
16 Acetonitrile	0.0657274	0.0570280	0.0532119	0.0535578	0.0500534	0.0495613		0.0548566		Avg 11
17 3-Chloro-1-propene	0.3532099	0.3149593	0.3180050	0.3100027	0.3090746	0.3073956		0.3187745		Avg 5.4
18 Methyl acetete	0.5753803	0.5361846	0.5035867	0.5397056	0.5326041	0.4971290		0.5307651		Avg 5.3
19 Methylene Chloride	0.6396227	0.6462756	0.5347180	0.5872436	0.5075793	0.5081112		0.5705917		Avg 11
20 Acrylonitrile	0.1602945	0.1454295	0.1448555	0.1374870	0.1380698	0.1305305		0.1427778		Avg 7.1
21 trans-1,2-Dichloroethe	0.7020121	0.6605596	0.5585906	0.6182791	0.5344388	0.5273541		0.6002057		Avg 12
22 Methyl tert-butyl ethe	1.5662873	1.5294594	1.2591142	1.3326065	1.1682677	1.1643493		1.3366807		Avg 13
23 Hexane	1.1109675	1.1605645	0.9812954	0.9971070	1.0441774	1.0239380		1.0530083		Avg 6.6
24 1,1-Dichloroethane	1.3914446	1.3847743	1.1504791	1.2699204	1.1445006	1.1526327		1.2489586		Avg 9.4
25 Vinyl acetate	0.5624626	0.5972505	0.6083726	0.5491670	0.6119226	0.5843734		0.5855914		Avg 4.3
26 Isopropyl ether	2.6978421	2.8216458	2.5156043	2.7883442	2.7894496	2.7425146		2.7259001		Avg 4.1
27 2-Chloro-1,3-butadiene	1.4104039	1.4795853	1.3201948	1.4541158	1.4659618	1.4419437		1.4287009		Avg 4.1
28 2,2-Dichloropropane	1.1255318	1.1058611	0.8771774	0.9803861	0.8163973	0.8102140		0.9525946		Avg 15
29 cis-1,2-Dichloroethene	0.6597344	0.7026977	0.5799984	0.6087217	0.5425856	0.5497544		0.6072487		Avg 10
30 2-Butanone (MEK)	0.2022518	0.1965617	0.2061988	0.1805322	0.1877483	0.1807350		0.1923380		Avg 5.7
31 Propionitrile	0.0350333	0.0328069	0.0346441	0.0312933	0.0307906	0.0310105		0.0325965		Avg 5.8
32 Ethyl acetate	0.2685895	0.2734375	0.2905668	0.2957470	0.2901179	0.2927163		0.2851959		Avg 4.0
33 Methacrylonitrile	0.2464108	0.2241458	0.2480020	0.2591721	0.2529364	0.2494790		0.2466910		Avg 4.8
34 Chlorobromomethane	0.2654237	0.2590992	0.2119624	0.2177522	0.2012682	0.2011993		0.2261175		Avg 13
35 Tetrahydrofuran	0.1421906	0.1238511	0.1263175	0.1166384	0.1072668	0.1132752		0.1215899		Avg 10
36 Chloroform	1.0692855	1.1327307	0.9403817	1.0314782	0.9110115	0.9248019		1.0016149		Avg 9.0
$ 37 Dibromofluoromethane		0.5974589	0.5291731	0.4983260	0.4807840	0.4592208		0.5129926		Avg 10
38 1,1,1-Trichloroethane	0.9864128	1.0955106	0.8597532	1.0017487	0.8448830	0.8899361		0.9463740		Avg 10
39 Cyclohexane	1.9165729	1.6782928	1.4101829	1.5304787	1.4971452	1.4898824		1.5870925		Avg 12
* 40 Pentafluorobenzene	491926	542545	633694	553833	643904	664038				
41 Carbon tetrachloride	0.5281422	0.5422307	0.4677081	0.5325973	0.4692543	0.5005792		0.5067520		Avg 6.5
42 1,1-Dichloropropene	0.9101526	0.9181067	0.7740066	0.8146801	0.7440675	0.7698701		0.8218139		Avg 9.1
$ 43 1,2-Dichloroethane-d4		0.4412949	0.4156537	0.3623634	0.3611186	0.3500415		0.3860944		Avg 10

(049-004)

Method: \\chi-svr07\chromdata\CMS06\20121002-13033.b\8260S-06.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
44 Isobutyl alcohol	0.0119079	0.0090083	0.0111179	0.0115094	0.0108184	0.0106619		0.0108373		Avg 9.3
45 Benzene	1.6023338	1.6434643	1.4562197	1.5116552	1.3693161	1.4365282		1.5032529		Avg 6.9
46 1,2-Dichloroethane	0.4860386	0.5197319	0.4720000	0.4673950	0.4399401	0.4632842		0.4747316		Avg 5.6
47 Isooctane	2.4501389	2.4302510	2.0509551	2.1441434	2.1631636	2.1625637		2.2335359		Avg 7.4
48 n-Heptane	0.8848286	0.8960866	0.7685161	0.7900773	0.7855289	0.7860593		0.8185161		Avg 6.9
* 49 1,4-Difluorobenzene	742197	825568	919147	828166	967033	985965				
50 n-Butanol	0.0049181	0.0044185	0.0051083	0.0054082	0.0054363	0.0052187		0.0050847		Avg 7.5
51 Trichloroethene	0.3618545	0.3859163	0.3293112	0.3458484	0.3116700	0.3283140		0.3438191		Avg 7.8
52 Methylcyclohexane	0.8958164	0.8890065	0.7413303	0.7819749	0.7439040	0.7228894		0.7958203		Avg 9.7
53 1,2-Dichloropropane	0.3906540	0.4191545	0.3574048	0.3719326	0.3429228	0.3575462		0.3732692		Avg 7.4
54 Dibromomethane	0.1786062	0.1941486	0.1645765	0.1634096	0.1507364	0.1545868		0.1676774		Avg 9.6
55 Methyl methacrylate	0.2620933	0.2350094	0.2427566	0.2415088	0.2215268	0.2193902		0.2370475		Avg 6.6
56 Dichlorobromomethane	0.4245884	0.4303048	0.3928236	0.4283547	0.3962810	0.4252868		0.4162732		Avg 4.1
57 2-Nitropropane	0.0415701	0.0451673	0.0610274	0.0686582	0.0748694	0.0741214	-20.93913	0.0736257		Linr 0.994
58 2-Chloroethyl vinyl et	0.0814322	0.0771838	0.0839081	0.0700128	0.0722970	0.0750735		0.0766512		Avg 6.9
59 cis-1,3-Dichloropropen	0.5077063	0.5001824	0.4612143	0.4845718	0.4508307	0.4756799		0.4800309		Avg 4.6
60 4-Methyl-2-pentanone (	0.3026606	0.2980921	0.3031316	0.2693128	0.2654465	0.2733167		0.2853267		Avg 6.2
$ 61 Toluene-d8 (Surr)		1.4956685	1.3891546	1.2983683	1.1985747	1.2146258		1.3192784		Avg 9.4
62 Toluene	0.9782365	0.9792036	0.8750167	0.9123693	0.8064706	0.8816497		0.9054911		Avg 7.3
63 trans-1,3-Dichloroprop	0.4180531	0.4556423	0.4138621	0.4278626	0.3892999	0.4291334		0.4223089		Avg 5.2
64 Ethyl methacrylate	0.3040355	0.2942290	0.3178648	0.2979984	0.2903430	0.2942231		0.2997823		Avg 3.3
65 1,1,2-Trichloroethane	0.3580588	0.3640586	0.3143404	0.3298569	0.2943634	0.3124429		0.3288535		Avg 8.3
66 Tetrachloroethene	0.4483618	0.4524085	0.4294052	0.4408802	0.4075990	0.4264557		0.4341851		Avg 3.8
67 1,3-Dichloropropane	0.4446279	0.4645930	0.4375079	0.4322704	0.4250350	0.4395765		0.4406018		Avg 3.1
68 2-Hexanone	0.2277408	0.2168933	0.2322403	0.2010948	0.2138769	0.2184830		0.2183882		Avg 5.0
69 Chlorodibromomethane	0.2176365	0.2484864	0.2414920	0.2719880	0.2670309	0.2858951		0.2554215		Avg 9.6
70 Ethylene Dibromide	0.2344158	0.2380913	0.2162216	0.2242134	0.1974302	0.2151040		0.2209127		Avg 6.7
* 71 Chlorobenzene-d5	719114	770698	819191	777657	822865	879550				
72 Chlorobenzene	1.1079095	1.1526384	1.0423675	1.0638035	0.9921933	1.0133176		1.0620383		Avg 5.6
73 1-Chlorohexane	0.8969665	0.8859953	0.7578095	0.8100916	0.7048718	0.7396563		0.7992318		Avg 9.9
74 1,1,1,2-Tetrachloroeth	0.3204993	0.3761418	0.3399899	0.3879774	0.3579807	0.3749497		0.3595898		Avg 7.1
75 Ethylbenzene	0.6432604	0.6647189	0.6067886	0.6217142	0.5804458	0.5911445		0.6180121		Avg 5.2
76 m-Xylene p-Xylene	1.7650013	1.8312901	1.6425366	1.7327243	1.6412187	1.6521697		1.7108235		Avg 4.6
77 o-Xylene	1.8298359	1.9255043	1.7107048	1.7873314	1.6824783	1.6881219		1.7706628		Avg 5.4
78 Styrene	1.2736730	1.3157954	1.1978099	1.2379466	1.1921574	1.1853512		1.2337889		Avg 4.2
79 Bromoform	0.1533306	0.1587583	0.1604072	0.1738820	0.1831885	0.1903389		0.1699843		Avg 8.7
80 Isopropylbenzene	3.9056363	3.9953245	3.7158130	3.7884399	3.6533406	3.8312201		3.8149624		Avg 3.3
81 Cyclohexanone	0.0992533	0.0918757	0.0985777	0.0850585	0.0878524	0.0778433		0.0900768		Avg 9.1
$ 82 4-Bromofluorobenzene (		0.7648958	0.7174118	0.6497302	0.6490864	0.6152138		0.6792676		Avg 8.9
84 Bromobenzene	0.8739088	0.8743371	0.8223868	0.8278052	0.7933680	0.8260933		0.8363165		Avg 3.8
85 1,1,2,2-Tetrachloroeth	0.5602924	0.5846205	0.6063212	0.5476078	0.5415949	0.5483154		0.5647920		Avg 4.5
86 1,2,3-Trichloropropane	0.1617195	0.1620180	0.1658613	0.1519641	0.1459577	0.1563374		0.1573097		Avg 4.7
87 trans-1,4-Dichloro-2-b	0.0862926	0.0859999	0.0997088	0.0965573	0.0916567	0.0901120		0.0917212		Avg 6.0
88 N-Propylbenzene	5.0935393	5.0542639	4.7705320	4.9423281	4.7739317	5.0422204		4.9461359		Avg 2.9
89 2-Chlorotoluene	3.1012417	3.0818863	2.8126396	2.8861248	2.7686211	2.9132748		2.9272981		Avg 4.7
90 1,3,5-Trimethylbenzene	3.4137719	3.3914283	3.1533845	3.2654391	3.0204296	3.0866367		3.2218484		Avg 5.0
91 4-Chlorotoluene	3.5021285	3.5203170	3.1906370	3.3278905	3.1113371	3.2050150		3.3095542		Avg 5.2
92 tert-Butylbenzene	2.7427150	2.9146385	2.6228874	2.7862194	2.6146053	2.7045011		2.7309278		Avg 4.1
93 Pentachloroethane	0.2627784	0.2420921	0.2792112	0.2600467	0.2637820	0.2761533		0.2640106		Avg 5.0
94 1,2,4-Trimethylbenzene	3.3737257	3.5125043	3.2116037	3.3600957	3.1392656	3.2576689		3.3091440		Avg 4.0
95 sec-Butylbenzene	4.4170988	4.5329458	4.1004635	4.3964043	4.1197085	4.2774064		4.3073379		Avg 4.0
96 1,3-Dichlorobenzene	1.7874388	1.8011913	1.6292647	1.6910776	1.6002159	1.6515818		1.6934617		Avg 4.9
97 4-Isopropyltoluene	3.6258089	3.7705591	3.4414898	3.6570194	3.4450806	3.5069825		3.5744901		Avg 3.7
* 98 1,4-Dichlorobenzene-d4	407587	450852	450890	449775	461688	474264				
99 1,4-Dichlorobenzene	1.8609197	1.7891254	1.6124730	1.6760460	1.6025360	1.6307019		1.6953003		Avg 6.3
100 n-Butylbenzene	3.8734766	3.8618570	3.4631864	3.5769719	3.3181560	3.3303790		3.5706712		Avg 7.0
101 1,2-Dichlorobenzene	1.5117907	1.5440597	1.4094211	1.3989383	1.3006947	1.2964175		1.4102204		Avg 7.3
104 1,2-Dibromo-3-Chloropr	0.0782270	0.0824766	0.0890298	0.0826163	0.0793940	0.0803745		0.0820197		Avg 4.7
107 1,3,5-Trichlorobenzene	1.2493795	1.3621468	1.1368434	1.1636890	1.1632292	1.0812236		1.1927519		Avg 8.3
108 1,2,4-Trichlorobenzene	0.9105076	0.9673267	0.8120988	0.7562995	0.6775518	0.6418638	0.5228610	0.8776057	-0.0012428	Quad 0.999
109 Hexachlorobutadiene	0.7026972	0.7298283	0.6006777	0.6192986	0.5497621	0.5340691		0.6227222		Avg 13

(049-005)

Method: \\chi-svr07\chromdata\CMS06\20121002-13033.b\8260S-06.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
110 Naphthalene	1.1792373	1.2908345	1.1808349	1.0227909	0.9538653	0.8945688		1.0870219		Avg 14
111 1,2,3-Trichlorobenzene	0.5630550	0.6163250	0.5119595	0.4428891	0.4097244	0.3702934	0.3485008	0.5508069	-0.0009376	Quad 0.998
112 2-Methylnaphthalene	0.1859049	0.2253214	0.1966617	0.1514446	0.2017160	Disabled		0.1922097		Avg 14

(049006)

Target and Internal Standards

Pentafluorobenzene
Acetone
Acrolein
Acrylonitrile
Bromochloromethane
Bromomethane
2-Butanone
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
Dichlorodifluoromethane
1,1-Dichloroethane
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
2,2-Dichloropropane
Iodomethane
Methylene chloride
1,1,1-Trichloroethane
Trichlorofluoromethane
Vinyl acetate
Vinyl Chloride
Dibromofluoromethane (surrogate)

Chlorobenzene-d_5
Bromoform
Bromofluorobenzene (surrogate)
Chlorodibromomethane
Chlorobenzene
1,3-Dichloropropane
Ethylbenzene
2-Hexanone
Styrene
1,1,1,2-Tetrachloroethane
Tetrachloroethene
Xylene

1,4-Difluorobenzene
Benzene
Bromodichloromethane
Carbon tetrachloride
2-Chloroethyl vinyl ether
1,2-Dibromoethane
Dibromomethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,2-Dichloropropane
1,1-Dichloropropene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
4-Methyl-2-pentanone
Toluene
Toluene-d_8 (surrogate)
1,1,2-Trichloroethane
Trichloroethene

1,4-Dichlorobenzene-d_4
Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
2-Chlorotoluene
4-Chlorotoluene
1,2-Dibromo-3-chloropropane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobutadiene
Isopropyl benzene
p-Isopropyltoluene
Naphthalene
n-Propylbenzene
1,1,2,2-Tetrachloroethane
1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

Attachment 3.

Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA- ICOC Form (052-001 to 052-005)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 112612_INST25 Worklist Num: 14007
Instrument: CMS25 Method: 8260W25c
Batch Directory: \\Chi-svr07\ChromData\CMS25\20121126-14007.b
Anaylysis Type: VOA Creator: Arndt, Brett D
Inj Volume: 5.00 Inj Vol Units: mL
Run Reagents:
8260 LOW IS_00103, Amount Added: 5.00 , Units: uL
8260 LOW SS_00105, Amount Added: 5.00 , Units: uL

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Fact	Client ID	Fraction
# 1 BFB	500-0014007-001	BFB		26-Nov-2012 07:36:30	25B1126.D	1	1.0		voaWater
# 2 CCV	500-0014007-002	CCV		26-Nov-2012 08:00:30	25C1126.D	2	1.0		voaWater
# 3 CCV IX	500-0014007-003	CCV		26-Nov-2012 08:24:30	25D1126.D	3	1.0		voaWater
# 4 LCS	500-0014007-004	LCS		26-Nov-2012 08:48:30	25S1126.D	4	1.0		voaWater
# 5 BLANK	500-0014007-005	Client		26-Nov-2012 09:12:30	BLA01.D	5	1.0		voaWater
# 6 MB	500-0014007-006	MB		26-Nov-2012 09:36:30	25M1126.D	6	1.0		voaWater
# 7 500-52523-B-1	500-0014007-007	Client		26-Nov-2012 10:01:30	52523-01.D	7	1.0	Storage Blank	voaWater
# 8 500-52552-B-30	500-0014007-008	Client		26-Nov-2012 10:25:30	52552-30.D	8	1.0	6155-EB-1	voaWater
# 9 500-52552-B-31	500-0014007-009	Client		26-Nov-2012 10:49:30	52552-31.D	9	1.0	6155-EB-2	voaWater
#10 500-52552-B-32	500-0014007-010	Client		26-Nov-2012 11:13:30	52552-32.D	10	1.0	6155-EB-3	voaWater
#11 500-52552-B-28	500-0014007-011	Client		26-Nov-2012 11:37:30	52552-28.D	11	2.0	6155-MW-DUP-2	voaWater
#12 500-52552-B-28	500-0014007-012	Client		26-Nov-2012 12:02:30	52552-28a.D	12	20.0	6155-MW-DUP-2	voaWater
#13 500-52552-B-29	500-0014007-013	Client		26-Nov-2012 12:26:30	52552-29.D	13	2.0	6155-MW-DUP-3	voaWater
#14 500-52552-B-29	500-0014007-014	Client		26-Nov-2012 12:50:30	52552-29a.D	14	20.0	6155-MW-DUP-3	voaWater
#15 LB 500-170750/1-A	500-0014007-015	LB		26-Nov-2012 13:14:30	LB-170750.D	15	20.0		voaWater
#16 720-46092-A-1-C	500-0014007-016	Client		26-Nov-2012 13:38:30	46092-01.D	16	20.0	F-REC-OIL-ECO11-5	voaWater
#17 500-52417-A-1-B	500-0014007-017	Client		26-Nov-2012 14:02:30	52417-01.D	17	20.0	F6-PDS-1-3.5	voaWater
#18 500-52417-A-2-B	500-0014007-018	Client		26-Nov-2012 14:26:30	52417-02.D	18	20.0	F6-PDS-2-3.5	voaWater
#19 500-52417-A-3-B	500-0014007-019	Client		26-Nov-2012 14:50:30	52417-03.D	19	20.0	F6-PDS-3.-3.5	voaWater
#20 500-52417-A-4-B	500-0014007-020	Client		26-Nov-2012 15:15:30	52417-04.D	20	20.0	F6-PDS-4-3.5	voaWater
#21 500-52417-A-5-B	500-0014007-021	Client		26-Nov-2012 15:39:30	52417-05.D	21	20.0	F6-PDS-5-3.5	voaWater
#22 500-52417-A-7-B	500-0014007-022	Client		26-Nov-2012 16:03:30	52417-07.D	22	20.0	F6-DUP-1	voaWater
#23 500-52417-A-8-B	500-0014007-023	Client		26-Nov-2012 16:27:30	52417-08.D	23	20.0	F6-PDS-6-3.5	voaWater
#24 500-52417-A-9-B	500-0014007-024	Client		26-Nov-2012 16:51:30	52417-09.D	24	20.0	F6-PDS-7-3.5	voaWater
#25 500-52417-A-10-B	500-0014007-025	Client		26-Nov-2012 17:15:30	52417-10.D	25	20.0	F6-PDF-1-4.5	voaWater
#26 500-52417-A-11-B	500-0014007-026	Client		26-Nov-2012 17:40:30	52417-11.D	26	20.0	F6-PDF-2-4.5	voaWater
#27 500-52417-A-12-B	500-0014007-027	Client		26-Nov-2012 18:04:30	52417-12.D	27	20.0	F6-PDS-8-3.5	voaWater
#28 500-52419-A-1-C	500-0014007-028	Client		26-Nov-2012 18:28:30	52419-01.D	28	20.0	WC-1	voaWater
#29 500-52419-A-1-C MS	500-0014007-029	MS		26-Nov-2012 18:52:30	52419-01s.D	29	20.0		voaWater
#30 500-52419-A-1-C MSD	500-0014007-030	MSD		26-Nov-2012 19:16:30	52419-01t.D	30	20.0		voaWater

(0 52-001)

Instrument ID# _____

TestAmerica Chicago
Corrective Action/Qualification Report GC/MS VOA and GC VOA

CHI-22-20-082/C-09/11

Analytical Methods
___SW846 8260B
___40CFR 624
___8015B
___8015C

CHROM Batch:_____
__Other_____
__WI_GRO
__OA1_GRO

Instrument Preventative Maintenance was performed per SOP_____

Tune Criteria
Description of Situation:_____
Action Taken:_____
Demonstration of Control:_____

Initial Calibration Criteria
Description of Situation:_____
Action Taken:_____
Demonstration of Control_____

ICV Criteria Eval _____

Continuing Calibration Criteria
Description of Situation:_____
Action Taken:_____
Demonstration of Control:_____

Internal Standards (CCV)
Description of situation:_____
Action Taken:_____
Demonstration of Control:_____

Method Blank
Description of situation:_____
Action Taken:_____
Demonstration of Control:_____

LCS
Description of Situation:_____
Action Taken:_____
Demonstration of Control:_____

Qualification of Data
Data Affected (Client/Sample #)_____
Qualification:_____
Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:_____

Client contacted/NCM # _____

Analyst Signature/date_____/_____

Reviewer Signature/date_____/_____

(052-002)

TestAmerica Chicago
GC/MS VOA Maintenance Logbook
Instrument No. 26

Page No._____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-Up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Date of Maintenance:_____ Entry No.:_____

Analyst:_____

Description:_____

Follow-Up:_____

Demonstration of Return to Control: Original Entry No: _____

Analyst:_____ Date:_____

Reviewer Signature or Initials / Date:_____ CHI-22-20-091/A-04/12

(052-003)

TestAmerica Chicago

GC/MS Volatiles: Sample Tracking Sheet

Screener: ______________ Job#

Sample File Name	Dilution	pH	Action	Inst./Date	Chrom Batch	MeOH Lot#	Analytical Batch	misc info

Reviewed by: ______________________ Date: ____________

CHI-22-20-075/D-0911

(052-004)

TestAmerica Chicago

GC/MS Volatiles: Internal Chain of Custody Form

ICOC Job Number:

Date	Time Out	Time In	ICOC Initials	misc info

CHI-22-20-074/A-01/08

(0520-005)

Attachment 4.

Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7) (053-001 to 053-008)

Report Date: 05-Dec-2012 11:15:07 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago
CCV, Cal Verification Report

Data File:	\\Chi-svr07\ChromData\CMS25\20121015-13236.b\25C1015p.D		
Lims ID:	CCV	Client ID:	
Inject. Date:	15-Oct-2012 21:40:30	Dil. Factor:	1.0000
Sample Type:	CCV		
Sample ID:	CCV		
Misc. Info.:	500-0013236-002		
Operator:	BDA	Instrument ID:	CMS25
Purge Vol:	5.000 mL	ALS Bottle#:	32
Lims Batch ID:	13236	Lims Sample ID:	2
Sublist:	chrom-8260W25c*sub3		
Raw Data:	Smoothed		
Detector:	MS SCAN		

Method:	\\Chi-svr07\ChromData\CMS25\20121015-13236.b\8260W25c.m		
Last Update:	16-Oct-2012 08:26:47	Calib Date:	09-Oct-2012 11:40:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\Chi-svr07\ChromData\CMS25\20121009-13144.b\25J1009h.D		
Limit Group:	MSVOA_8260_ICAL_WATER		
Integrator:	RTE	ID Type:	RT Order ID
Column Type:		Column Dia:	
Process Host:	XAWRK012		

First Level Reviewer: manzanol Date: 15-Oct-2012 23:26:39

Start Cal Date: 26-Sep-2012 08:40:30
End Cal Date: 09-Oct-2012 11:40:30

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
1 Dichlorodifluorometha	0.480698	0.000		0.436364	0.010	-9.2	50.0
2 Chloromethane	0.623198	0.000		0.521937	0.100	-16.2	50.0
3 Vinyl chloride	0.613386	0.000		0.555394	0.010	-9.5	20.0
5 Bromomethane	0.234468	0.000		0.225947	0.010	-3.6	50.0
6 Chloroethane	0.493999	0.000		0.564098	0.010	14.2	50.0
7 Trichlorofluoromethan	0.889626	0.000		0.831525	0.010	-6.5	50.0
8 Dichlorofluoromethane	1.005352	0.000		0.934303	0.010	-7.1	50.0
10 1,1,2-Trichloro-1,2,2	0.499449	0.000		0.471231	0.010	-5.6	50.0
11 Acrolein	0.036173	0.000		0.031121	0.001	-14.0	50.0
12 1,1-Dichloroethene	0.393610	0.000		0.365621	0.010	-7.1	20.0
13 Acetone	50.0	0.000	48.5	0.044005	0.010	-3.1	50.0
15 Carbon disulfide	1.322184	0.000		1.268366	0.010	-4.1	50.0
18 Acetonitrile	400.0	0.000	394.9	0.019178	0.001	-1.3	50.0
19 Methylene Chloride	0.349638	0.000		0.342101	0.010	-2.2	50.0
20 Methyl tert-butyl eth	0.507774	0.000		0.467992	0.010	-7.8	50.0
21 trans-1,2-Dichloroeth	0.432109	0.000		0.413216	0.010	-4.4	50.0
22 Acrylonitrile	0.064080	0.000		0.059764	0.001	-6.7	50.0

(053-001)

Report Date: 05-Dec-2012 11:15:07 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\25C1015p.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
25 1,1-Dichloroethane	0.821773	0.000		0.792714	0.100	-3.5	50.0
26 Vinyl acetate	0.259013	0.000		0.297764	0.010	15.0	50.0
28 2,2-Dichloropropane	0.587908	0.000		0.586974	0.010	-0.2	50.0
29 cis-1,2-Dichloroethen	0.429541	0.000		0.427265	0.010	-0.5	50.0
30 2-Butanone (MEK)	0.058949	0.000		0.056303	0.010	-4.5	50.0
32 Propionitrile	0.016555	0.000		0.016217	0.001	-2.0	50.0
34 Tetrahydrofuran	0.038811	0.000		0.032574	0.010	-16.1	50.0
33 Chlorobromomethane	0.165548	0.000		0.172211	0.010	4.0	50.0
36 Chloroform	0.670929	0.000		0.666966	0.010	-0.6	20.0
38 1,1,1-Trichloroethane	0.671233	0.000		0.680649	0.010	1.4	50.0
$ 39 Dibromofluoromethane	0.362406	0.000		0.364034	0.010	0.4	50.0
41 Carbon tetrachloride	0.531365	0.000		0.537685	0.010	1.2	50.0
42 1,1-Dichloropropene	0.625901	0.000		0.572143	0.010	-8.6	50.0
45 Benzene	1.205620	0.000		1.179109	0.010	-2.2	50.0
$ 46 1,2-Dichloroethane-d4	0.245518	0.000		0.237279	0.010	-3.4	50.0
47 1,2-Dichloroethane	0.317854	0.000		0.310824	0.010	-2.2	50.0
50 Trichloroethene	0.370778	0.000		0.387501	0.010	4.5	50.0
53 1,2-Dichloropropane	0.305481	0.000		0.309489	0.010	1.3	20.0
55 Dibromomethane	0.120498	0.000		0.123898	0.010	2.8	50.0
56 Dichlorobromomethane	0.341551	0.000		0.351045	0.010	2.8	50.0
57 2-Chloroethyl vinyl e	0.088749	0.000		0.086851	0.010	-2.1	50.0
59 cis-1,3-Dichloroprope	0.350974	0.000		0.359758	0.010	2.5	50.0
60 4-Methyl-2-pentanone	0.093690	0.000		0.091132	0.010	-2.7	50.0
$ 61 Toluene-d8 (Surr)	1.108916	0.000		1.104413	0.010	-0.4	50.0
62 Toluene	0.775776	0.000		0.805678	0.010	3.9	20.0
63 trans-1,3-Dichloropro	0.276699	0.000		0.279547	0.010	1.0	50.0
65 1,1,2-Trichloroethane	0.200593	0.000		0.213122	0.010	6.2	50.0
66 Tetrachloroethene	0.499889	0.000		0.520293	0.010	4.1	50.0
67 1,3-Dichloropropane	0.320185	0.000		0.325097	0.010	1.5	50.0
68 2-Hexanone	0.070377	0.000		0.067366	0.010	-4.3	50.0
69 Chlorodibromomethane	0.257038	0.000		0.286692	0.010	11.5	50.0
70 Ethylene Dibromide	0.146479	0.000		0.161688	0.010	10.4	50.0
71 1-Chlorohexane	0.629198	0.000		0.613031	0.010	-2.6	50.0
73 Chlorobenzene	0.994133	0.000		1.030253	0.300	3.6	50.0
74 Ethylbenzene	0.587174	0.000		0.615665	0.010	4.9	20.0
75 1,1,1,2-Tetrachloroet	0.340371	0.000		0.371346	0.010	9.1	50.0

(053-002)

Report Date: 05-Dec-2012 11:15:07 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\25C1015p.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
76 m-Xylene p-Xylene	1.366159	0.000		1.398027	0.010	2.3	50.0
77 o-Xylene	1.303301	0.000		1.356544	0.010	4.1	50.0
78 Styrene	0.993286	0.000		1.102584	0.010	11.0	50.0
80 Bromoform	0.149674	0.000		0.168837	0.100	12.8	50.0
81 Isopropylbenzene	3.633320	0.000		3.925434	0.010	8.0	50.0
$ 83 4-Bromofluorobenzene	0.405511	0.000		0.392671	0.010	-3.2	50.0
84 Bromobenzene	0.790581	0.000		0.886507	0.010	12.1	50.0
85 1,1,2,2-Tetrachloroet	0.350689	0.000		0.369221	0.300	5.3	50.0
86 N-Propylbenzene	4.252096	0.000		4.425071	0.010	4.1	50.0
87 1,2,3-Trichloropropan	0.284829	0.000		0.289531	0.010	1.7	50.0
89 2-Chlorotoluene	2.288261	0.000		2.413620	0.010	5.5	50.0
90 1,3,5-Trimethylbenzen	2.854604	0.000		3.121315	0.010	9.3	50.0
91 4-Chlorotoluene	2.417443	0.000		2.457764	0.010	1.7	50.0
92 tert-Butylbenzene	2.658628	0.000		2.980217	0.010	12.1	50.0
94 1,2,4-Trimethylbenzen	2.784006	0.000		3.015182	0.010	8.3	50.0
95 sec-Butylbenzene	4.081124	0.000		4.417756	0.010	8.2	50.0
96 1,3-Dichlorobenzene	1.678329	0.000		1.754500	0.010	4.5	50.0
97 4-Isopropyltoluene	3.435029	0.000		3.717667	0.010	8.2	50.0
99 1,4-Dichlorobenzene	1.624419	0.000		1.670596	0.010	2.8	50.0
100 n-Butylbenzene	3.128006	0.000		3.095475	0.010	-1.0	50.0
101 1,2-Dichlorobenzene	1.331469	0.000		1.452888	0.010	9.1	50.0
102 1,2-Dibromo-3-Chlorop	0.046811	0.000		0.052635	0.010	12.4	50.0
104 1,2,4-Trichlorobenzen	0.919954	0.000		0.923145	0.010	0.3	50.0
105 Hexachlorobutadiene	0.807587	0.000		0.867167	0.010	7.4	50.0
106 Naphthalene	1.044830	0.000		1.129597	0.010	8.1	50.0
107 1,2,3-Trichlorobenzen	0.736654	0.000		0.774832	0.010	5.2	50.0
S 109 Trimethylbenzene, Tot			108.8				50.0
S 110 Trihalomethanes, Tota			213.3				50.0
S 112 1,3-Dichloropropene,			101.8				50.0
S 113 Xylenes, Total	150.0		154.4			2.9	50.0
S 117 1,2-Dichloroethene, T	100.0		97.5			-2.5	50.0

(053-003)

Report Date: 05-Dec-2012 11:16:02 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago
CCV, Cal Verification Report

Data File:	\\Chi-svr07\ChromData\CMS25\20121015-13236.b\25D1015p.D		
Lims ID:	CCV IX	Client ID:	
Inject. Date:	15-Oct-2012 22:04:30	Dil. Factor:	1.0000
Sample Type:	CCV		
Sample ID:	CCV IX		
Misc. Info.:	500-0013236-003		
Operator:	BDA	Instrument ID:	CMS25
Purge Vol:	5.000 mL	ALS Bottle#:	33
Lims Batch ID:	13236	Lims Sample ID:	3
Sublist:	chrom-8260W25c*sub5		
Raw Data:	Smoothed		
Detector:	MS SCAN		
Method:	\\Chi-svr07\ChromData\CMS25\20121015-13236.b\8260W25c.m		
Last Update:	16-Oct-2012 08:26:47	Calib Date:	09-Oct-2012 11:40:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\Chi-svr07\ChromData\CMS25\20121009-13144.b\25J1009h.D		
Limit Group:	MSVOA_8260_ICAL_WATER		
Integrator:	RTE	ID Type:	RT Order ID
Column Type:		Column Dia:	
Process Host:	XAWRK012		
First Level Reviewer:	manzanol	Date:	15-Oct-2012 23:27:02
Start Cal Date:	26-Sep-2012 08:40:30		
End Cal Date:	09-Oct-2012 11:40:30		

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
4 Butadiene	0.598018	0.000		0.631605	0.010	5.6	50.0
9 Ethyl ether	0.229263	0.001		0.236476	0.010	3.1	50.0
14 Iodomethane	100.0	0.000	80.3	0.460925	0.010	-19.7	50.0
16 3-Chloro-1-propene	0.220239	0.000		0.217617	0.010	-1.2	50.0
17 Methyl acetate	50.0	0.000	56.8	0.154086	0.010	13.5	50.0
23 Hexane	0.808078	0.001		0.868453	0.010	7.5	50.0
24 Isopropyl ether	1.019772	0.000		1.157572	0.010	13.5	50.0
27 2-Chloro-1,3-butadien	0.775379	0.000		0.865838	0.010	11.7	50.0
31 Ethyl acetate	0.131561	0.000		0.138201	0.010	5.0	50.0
35 Methacrylonitrile	0.092576	0.001		0.094965	0.010	2.6	50.0
37 Cyclohexane	0.884799	0.000		0.967591	0.010	9.4	50.0
$ 39 Dibromofluoromethane	0.362406	0.000		0.363322	0.010	0.3	50.0
43 Isobutyl alcohol	0.006728	0.000		0.007647	0.001	13.7	50.0
44 Isooctane	1.635131	0.000		1.825340	0.010	11.6	50.0
$ 46 1,2-Dichloroethane-d4	0.245518	0.000		0.244520	0.010	-0.4	50.0
48 n-Heptane	0.623122	0.000		0.685015	0.010	9.9	50.0
51 n-Butanol	4000.0	0.005	4115.1	0.002157	0.001	2.9	50.0

(053-004)

Report Date: 05-Dec-2012 11:16:02 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\Chi-svr07\ChromData\CMS25\20121015-13236.b\25D1015p.D

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
52 Methylcyclohexane	0.574944	0.001		0.640881	0.010	11.5	50.0
54 Methyl methacrylate	0.106481	0.000		0.113944	0.010	7.0	50.0
58 2-Nitropropane	0.045730	0.000		0.050395	0.010	10.2	50.0
$ 61 Toluene-d8 (Surr)	1.108916	0.000		1.091314	0.010	-1.6	50.0
64 Ethyl methacrylate	0.178146	-0.005		0.199618	0.010	12.1	50.0
82 Cyclohexanone	0.021551	0.000		0.020655	0.010	-4.2	50.0
$ 83 4-Bromofluorobenzene	0.405511	0.000		0.425770	0.010	5.0	50.0
88 trans-1,4-Dichloro-2-	0.050920	0.000		0.056149	0.010	10.3	50.0
93 Pentachloroethane	0.264805	0.000		0.287522	0.010	8.6	50.0
103 1,3,5-Trichlorobenzen	50.0	0.000	55.2	1.375048	0.010	10.4	50.0
108 2-Methylnaphthalene	50.0	0.000	46.6	0.571690	0.010	-6.9	50.0

(053-005)

Report Date: 05-Dec-2012 11:04:03 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

TestAmerica Chicago
CCV, Cal Verification Report

Data File:	\\chi-svr07\chromdata\CMS06\20121002-13033.b\6C1002.d		
Lims ID:	CCV	Client ID:	
Inject. Date:	02-Oct-2012 14:42:30	Dil. Factor:	1.0000
Sample Type:	CCV		
Sample ID:	CCV		
Misc. Info.:	500-0013033-005		
Operator:	WBS	Instrument ID:	CMS06
Purge Vol:	5.000 mL	ALS Bottle#:	2
Lims Batch ID:	13033	Lims Sample ID:	5
Sublist:	chrom-8260S-06*sub2		
Detector:	MS SCAN		
Method:	\\chi-svr07\chromdata\CMS06\20121002-13033.b\8260S-06.m		
Method Label:	TAL Chicago VOA Report SW846 8260B		
Last Update:	03-Oct-2012 11:37:20	Calib Date:	01-Oct-2012 22:06:30
Quant Method:	Internal Standard	Quant By:	Initial Calibration
Last ICal File:	\\chi-svr07\chromdata\CMS06\20121001-13011.b\6J1001f.d		
Limit Group:	MSVOA_8260_ICAL_SOIL_LOW		
Integrator:	RTE	ID Type:	RT Order ID
Column Type:		Column Dia:	
Process Host:	XAWRK012		
First Level Reviewer: segallyb		Date:	02-Oct-2012 15:02:58
Start Cal Date:	01-Oct-2012 17:56:30		
End Cal Date:	01-Oct-2012 22:06:30		

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
1 Dichlorodifluorometha	0.968933	0.000		0.980264	0.010	1.2	50.0
2 Chloromethane	1.168112	0.006		1.212284	0.100	3.8	50.0
3 Vinyl chloride	0.899908	0.006		0.982244	0.010	9.1	20.0
5 Bromomethane	0.198855	0.009		0.216388	0.010	8.8	50.0
6 Chloroethane	0.321482	-0.006		0.341919	0.010	6.4	50.0
7 Dichlorofluoromethane	1.157316	-0.006		1.302592	0.010	12.6	50.0
8 Trichlorofluoromethan	0.960609	0.006		1.086451	0.010	13.1	50.0
10 Acrolein	0.077348	-0.003		0.085630	0.001	10.7	50.0
11 1,1-Dichloroethene	0.549813	0.003		0.642478	0.010	16.9	20.0
12 1,1,2-Trichloro-1,2,2	0.695016	0.014		0.787043	0.010	13.2	50.0
13 Acetone	0.173372	0.000		0.199013	0.010	14.8	50.0
15 Carbon disulfide	2.171632	0.006		2.443601	0.010	12.5	50.0
16 Acetonitrile	0.054857	0.000		0.057611	0.001	5.0	50.0
19 Methylene Chloride	0.570592	0.002		0.626243	0.010	9.8	50.0
20 Acrylonitrile	0.142778	0.003		0.143036	0.001	0.2	50.0
21 trans-1,2-Dichloroeth	0.600206	0.003		0.677657	0.010	12.9	50.0
22 Methyl tert-butyl eth	1.336681	0.003		1.397253	0.010	4.5	50.0

(053-006)

Report Date: 05-Dec-2012 11:04:03 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS06\20121002-13033.b\6C1002.d

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
24 1,1-Dichloroethane	1.248959	0.000		1.351409	0.100	8.2	50.0
25 Vinyl acetate	0.585591	0.003		0.534573	0.010	-8.7	50.0
28 2,2-Dichloropropane	0.952595	0.000		1.064153	0.010	11.7	50.0
29 cis-1,2-Dichloroethen	0.607249	0.000		0.659702	0.010	8.6	50.0
30 2-Butanone (MEK)	0.192338	-0.002		0.216371	0.010	12.5	50.0
31 Propionitrile	0.032596	0.000		0.033610	0.001	3.1	50.0
34 Chlorobromomethane	0.226117	0.000		0.241431	0.010	6.8	50.0
35 Tetrahydrofuran	0.121590	0.006		0.123609	0.010	1.7	50.0
36 Chloroform	1.001615	0.000		1.070006	0.010	6.8	20.0
$ 37 Dibromofluoromethane	0.512993	0.006		0.463203	0.010	-9.7	50.0
38 1,1,1-Trichloroethane	0.946374	-0.002		1.070729	0.010	13.1	50.0
41 Carbon tetrachloride	0.506752	-0.008		0.582267	0.010	14.9	50.0
42 1,1-Dichloropropene	0.821814	-0.006		0.869588	0.010	5.8	50.0
$ 43 1,2-Dichloroethane-d4	0.386094	0.008		0.339040	0.010	-12.2	50.0
45 Benzene	1.503253	0.003		1.620705	0.010	7.8	50.0
46 1,2-Dichloroethane	0.474732	-0.003		0.499492	0.010	5.2	50.0
51 Trichloroethene	0.343819	0.000		0.381598	0.010	11.0	50.0
53 1,2-Dichloropropane	0.373269	-0.005		0.387036	0.010	3.7	20.0
54 Dibromomethane	0.167677	0.000		0.176141	0.010	5.0	50.0
56 Dichlorobromomethane	0.416273	0.000		0.439234	0.010	5.5	50.0
58 2-Chloroethyl vinyl e	0.076651	0.000		0.076787	0.010	0.2	50.0
59 cis-1,3-Dichloroprope	0.480031	-0.003		0.504191	0.010	5.0	50.0
60 4-Methyl-2-pentanone	0.285327	0.000		0.289993	0.010	1.6	50.0
$ 61 Toluene-d8 (Surr)	1.319278	0.000		1.243074	0.010	-5.8	50.0
62 Toluene	0.905491	0.000		0.970063	0.010	7.1	20.0
63 trans-1,3-Dichloropro	0.422309	0.000		0.440982	0.010	4.4	50.0
65 1,1,2-Trichloroethane	0.328854	-0.003		0.329008	0.010	0.0	50.0
66 Tetrachloroethene	0.434185	0.000		0.470565	0.010	8.4	50.0
67 1,3-Dichloropropane	0.440602	0.000		0.433026	0.010	-1.7	50.0
68 2-Hexanone	0.218388	0.003		0.224503	0.010	2.8	50.0
69 Chlorodibromomethane	0.255421	0.003		0.260235	0.010	1.9	50.0
70 Ethylene Dibromide	0.220913	0.000		0.228101	0.010	3.3	50.0
72 Chlorobenzene	1.062038	0.000		1.125134	0.300	5.9	50.0
73 1-Chlorohexane	0.799232	0.000		0.890016	0.010	11.4	50.0
74 1,1,1,2-Tetrachloroet	0.359590	-0.003		0.389060	0.010	8.2	50.0
75 Ethylbenzene	0.618012	0.002		0.658827	0.010	6.6	20.0

(053007)

Report Date: 05-Dec-2012 11:04:03 Chrom Revision: 2.0 22-Oct-2012 16:30:48

Preliminary Report

Data File: \\chi-svr07\chromdata\CMS06\20121002-13033.b\6C1002.d

Compound	Standard RRF/Amount	DLT RT	Ccal Amount	Ccal RF	Min. RRF	%D	Max. %D
76 m-Xylene p-Xylene	1.710823	-0.003		1.863721	0.010	8.9	50.0
77 o-Xylene	1.770663	0.000		1.909514	0.010	7.8	50.0
78 Styrene	1.233789	0.000		1.346082	0.010	9.1	50.0
79 Bromoform	0.169984	0.000		0.174803	0.100	2.8	50.0
80 Isopropylbenzene	3.814962	0.000		4.048226	0.010	6.1	50.0
$ 82 4-Bromofluorobenzene	0.679268	0.000		0.610222	0.010	-10.2	50.0
84 Bromobenzene	0.836317	0.002		0.866699	0.010	3.6	50.0
85 1,1,2,2-Tetrachloroet	0.564792	0.000		0.613506	0.300	8.6	50.0
86 1,2,3-Trichloropropan	0.157310	-0.003		0.165097	0.010	5.0	50.0
88 N-Propylbenzene	4.946136	-0.001		5.251128	0.010	6.2	50.0
89 2-Chlorotoluene	2.927298	0.000		3.063517	0.010	4.7	50.0
90 1,3,5-Trimethylbenzen	3.221848	0.000		3.467587	0.010	7.6	50.0
91 4-Chlorotoluene	3.309554	-0.003		3.580859	0.010	8.2	50.0
92 tert-Butylbenzene	2.730928	0.003		2.956293	0.010	8.3	50.0
94 1,2,4-Trimethylbenzen	3.309144	0.000		3.552070	0.010	7.3	50.0
95 sec-Butylbenzene	4.307338	0.003		4.614123	0.010	7.1	50.0
96 1,3-Dichlorobenzene	1.693462	0.000		1.828970	0.010	8.0	50.0
97 4-Isopropyltoluene	3.574490	0.000		3.905785	0.010	9.3	50.0
99 1,4-Dichlorobenzene	1.695300	0.000		1.809905	0.010	6.8	50.0
100 n-Butylbenzene	3.570671	0.000		3.946256	0.010	10.5	50.0
101 1,2-Dichlorobenzene	1.410220	0.000		1.542207	0.010	9.4	50.0
104 1,2-Dibromo-3-Chlorop	0.082020	0.003		0.094586	0.010	15.3	50.0
108 1,2,4-Trichlorobenzen	50.0	0.000	55.2	0.903601	0.010	10.4	50.0
109 Hexachlorobutadiene	0.622722	0.000		0.684011	0.010	9.8	50.0
110 Naphthalene	1.087022	-0.001		1.204724	0.010	10.8	50.0
111 1,2,3-Trichlorobenzen	50.0	0.000	54.4	0.550920	0.010	8.8	50.0
S 113 1,2-Dichloroethene, T	100.0		110.8			10.8	50.0
S 114 Xylenes, Total	150.0		162.9			8.6	50.0
S 115 1,3-Dichloropropene,			104.7				50.0
S 116 Trihalomethanes, Tota			208.5				50.0
S 117 Trimethylbenzene, Tot			216.0				50.0

(053008)

Attachment 5.

Example: Analysis and Sample Tracking Flowcharts

Analysis Scheme Flowchart

(Terms defined in the Section 9)

```
                PFTBA
                  |
                  |
                  |
                 BFB  (Form 5)
                /  \  Every 12 hours
               /    \
              /      \
Initial Calibration--------Continuing Calibration
   (Form 6)                  (Form 7)
                          Every 12 hours
                              |
                              |
                             \|/
                           Analysis
                              |
                              |
                             \|/
                 Computer Data Production/Reduction
                              |
                              |
                             \|/
                 Operator Data Review/Reduction
                      (Form 1,2,3,4,5)
                              |
                              |
                             \|/
                  Final Review/Data Submission
                              |
                              |
                             \|/
                       Report Generation
```

Sample Tracking Flowchart

Sample Receipt
| \
| \
Log-In VOA Refrigerator
| |
COC |
| |
Computer |
| |
| |
| |
Backlog |
| |
| /
| /
| /
| /
VOAs
|
|
INPUT
|
Sample Tracking Board, Sample Tracking Sheets
|
Analysis
|
OUTPUT: COC, Analysis Logbook, Sample
Data, Tune, Calibration, QC, Narrative/NCM
|

· Operator Review/Data Reduction · Archival (Automated scheduled back-ups)
· Data Package w/ Review Form, CN · Data purged off system
· 1st 100% Review
· Submission/Final Review
|
REPORT GENERATION
· Data Package, Misc Files scanned
· Review Form filed

Attachment 6.

Example: Data Review Checklist (057-001 to 057-003)

TestAmerica
THE LEADERS IN ENVIRONMENTAL TESTING

Chicago
CHI-22-20-088/D-09/12

GC/MS Initial Calibration Review Checklist

Method: _____
Instrument: _____
Worklist Number or Batch: _____
Analysis Date: _____

Item for Review	Level 1	Level 2
TUNE:		
Tune passes, all points within the 12 hour clock?		
Reports:		
p-Flag reports reviewed and accepted?		
ICAL Pass / Fail reviewed and accepted?		
Average/Short Form 6 reviewed?		
Compounds:		
Linearity: CCC's ≤ 30% RSD		
Linear or Quadratic Regression curve fit for all >15% RSD; 20% RSD -8270D; R^2 >0.990		
Plots for all Linear Regressions reviewed?		
conc-intercept < RL (<1/2 RL DoD QSM); < 30% Error (Chrom: Details of Calibration per Analyte)		
Isomeric Pairs reviewed and accepted? (Chrom: View/Documents/Methods/Isomer)		
Responses: SPCCs all pass minimum response factors?		
All manual integrations reviewed were found to be necessary, were performed correctly and have documented reasons?		
ICAL selection of points – documented & performed correctly		
Method:		
Method locked in Chrom?		
Method set as most recent?		
Method uploaded to TALs?		
Calibration events correct and locked in TALs?		
Initial Calibration Verification (ICV):		
ICV performed and evaluated against 70-130% (30%D) criteria? AFCEE-75-125% (25%D)		
QAPP Specific ICV 80-120% (20%D) criteria?		
Comments:		

Note: By signing below I agree that I have reviewed the data as indicated on this checklist and according to the acceptance criteria found in the applicable SOP.

Primary Reviewer: _____ Date _____

Secondary Reviewer: _____ Date _____

(057-001)

TestAmerica Chicago
GCMS Volatiles DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Method: a) 8260B_____ 624 _____ 5030_____ Encores: 5035--High _____ 5035-Low_____ TCLP _____

Matrix: WATER / SOIL / SPLP-TCLP / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Inst# Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: Calib. ID: _____ _____ _____ _____ _____ _____
Sample Hold Time: _____ NCM Ref Number: _____			Date Analyzed: _____
Data Summaries: Executive Summary Are all flags correctly assigned? Y N Sample Data Sheets present? Y N Surrogate page present? Y N QC Data Summaries all present? Y N QC Associated Summary All samples associated? Y N Are 5035 samples linked to prep batch? Y N NA Are all dilutions appearing on summary? Y N NA			
FORM 1: IF **original** and **re-run** are to be reported in LIMS: Appropriate suffixes present Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Smp # Original Dilution Comments
FORM 2: Surrogate Recoveries Within Limits Statistical Limits ____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits ____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits ____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

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TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Limits Met (< 1/2 RL for AFCEE / DoD QSM) NCM Ref Number: _____			
FORM 5: Tuning Criteria Met/Matches LIMS Analysis Batches ICAL Form 5 OK? Yes No Tunes: _____ _____ _____ _____ _____ _____ _____ _____ _____ NCM Ref Number: _____			Tune time met? Yes No
FORM 8: Internal Standards Criteria Met MPIS or CCVIS NCM Ref Number: _____			List sample numbers:
RAW DATA: 1) Raw Data Verified/Complete			
2) Screening Data match analysis data?			
Form 6: Initial Calibration Criteria met and Complete?			
Form 7: CCV criteria met method criteria NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Tune Yes No 2) Blank Yes No 3) LCS/LCSD Yes No 4) MS/MSD Yes No NA MRL Check Required: Yes No Control Limit Applied: _____			
Manual Integration reports (before and afters) present, analyst initials are present and the reasons for the MI are correctly documented and approved?			
Prep Log page Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required/Properly Documented Yes No			
Additional Comments:			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : _____ Compound: _____

(057-003)

Attachment 7.

CA-T-P-002: TestAmerica Corporate Policy for the Selection of Calibration Points (058-001 to 058-004)

Title: Selection of Calibration Points

Approvals (Signature/Date):

4/13/2011	4/1/2011
Richard Burrows — Date	Dr. Charles W. Carter — Date
Director of Technical Services	VP, Quality & Technical Services

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1.0 PURPOSE

This policy describes TestAmerica's requirements for determination of the number of points, and removal of points from calibration curves.

2.0 SCOPE

This Policy applies to all multi-level initial calibrations.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

5.0 PROCEDURE

5.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved Standard Operating Procedure (SOP) that is what will be used.

5.2 In the cases not defined in Section 5.1, the number of data points will be determined by the technical manager based on the Data Quality Objectives (DQOs) for precision and accuracy to be met by the method.

5.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2 Non-detects may use a simple point at the reporting level.

Examples:

A. Need to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by UV-HPLC at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the

(058-002)

recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the data quality objectives will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in laboratory for meeting regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A five-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the Quality Assurance (QA) Manager.

5.3 Removal of Points from a Calibration Curve

5.3.1 Removal or replacement of levels from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24 hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internals show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).
- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (all analytes in spike mix must

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show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection)

5.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers) the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

All TestAmerica associates utilizing methods involving multi-point calibrations are required to follow this policy.

7.0 REFERENCES/CROSS-REFERENCES

None.

8.0 ATTACHMENTS

None.

9.0 REVISION HISTORY

- Revision 0, dated 20 November 2007
  - Initial Release. Previously Policy No. P-T-001.
- Revision 1, dated 23 June 2009.
  - No Changes.
- Revision 2, dated 1 April 2011.
  - Secs. 5.2.1-5.2.2: Section added to address the minimum number of calibration points as cited by the 2009 TNI Standard.

(058-004)

Attachment 8.

CA-Q-S-002: TestAmerica Corporate SOP for Manual Integrations (059-001 to 059-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

08/01/2012	08/01/2012
Raymond J. Frederici Quality and EH&S Director	Larry Penfold Quality Compliance Director
Date	Date

08/01/2012
Verl D. Preston
Quality Assessment Director
Date

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1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

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4.5 **Baseline:** The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 **Peak:** An increase in signal from baseline to a maximum and then back to baseline.

4.7 **Coelution:** The concurrent elution of two or more compounds to the detector at the same time.

4.8 **Elution:** The process of movement of compounds from the chromatographic system.

4.9 **Carry-over:** Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 **Peak Tailing:** Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 **Interference Peaks or Fused Peaks:** Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important considerations in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

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5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters is necessary to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

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5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibrations standards, QC standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices. This

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process for before & after chromatograms is also required for OVAP-approved laboratories, unless instructed otherwise.

5.4.7.1 Re-integration technique marked, if available, on the data system.

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

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5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

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5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.

(059-008)

- Expanded section on data miner software to include Chrom Audit Miner
- Included reference to data miner training presentation.

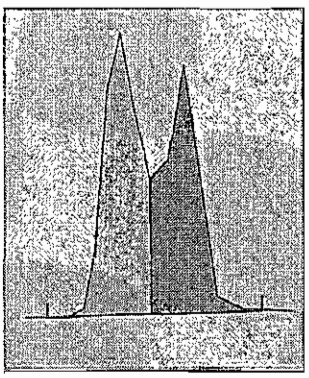
- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples

(059-009)

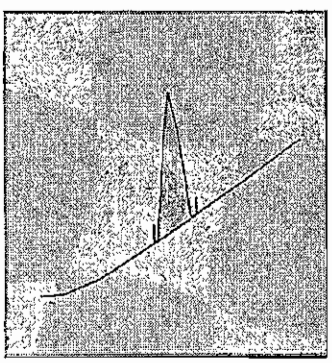
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

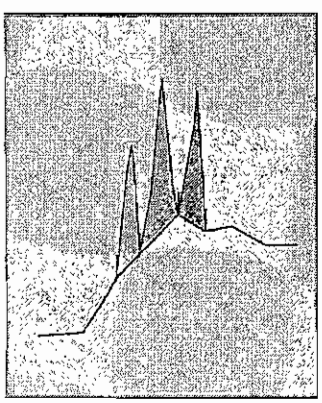
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.



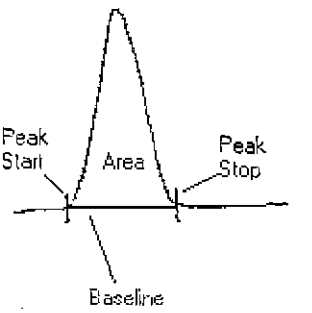
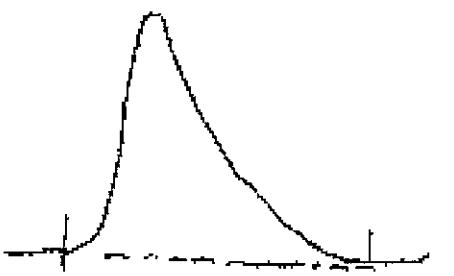
Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.



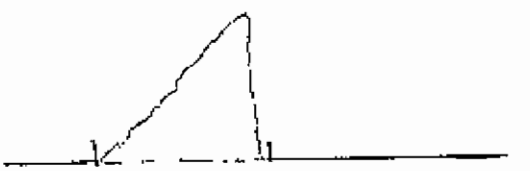
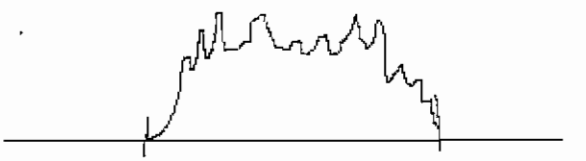
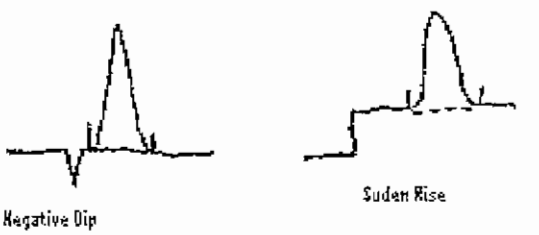
Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.



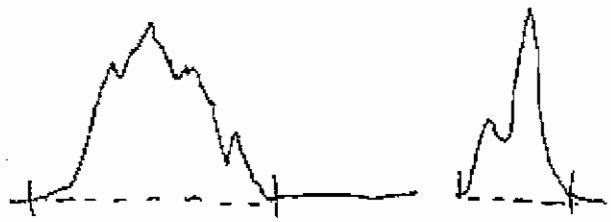
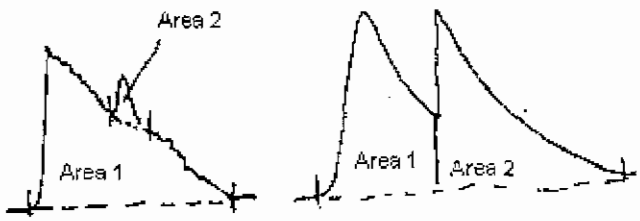
(059-010)

Ideal peak (Gaussian Shape) The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

(059-011)

Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	

(059-012)

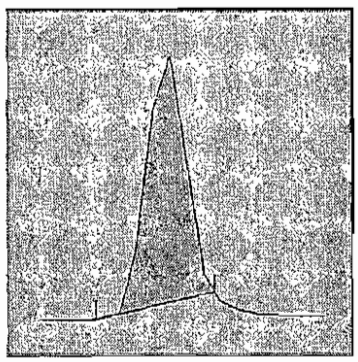
Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

(059-013)

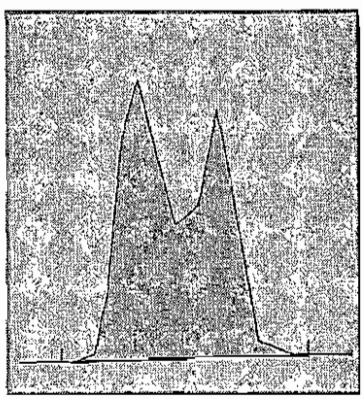
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

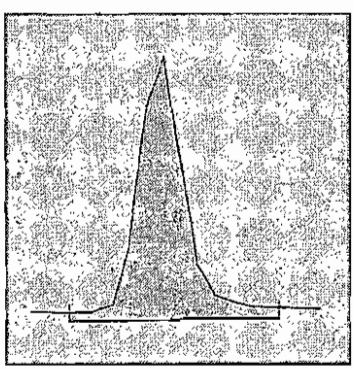
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.

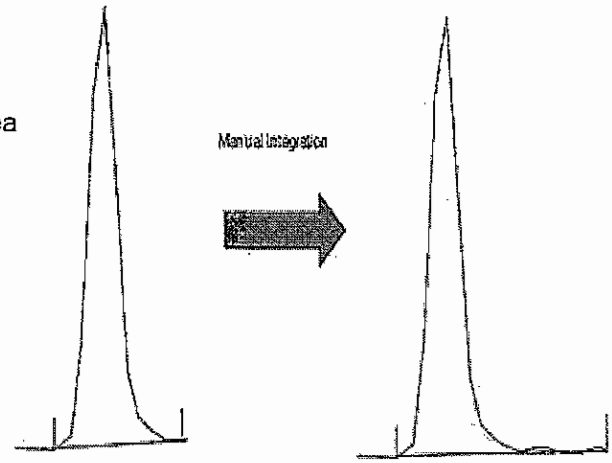


Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.

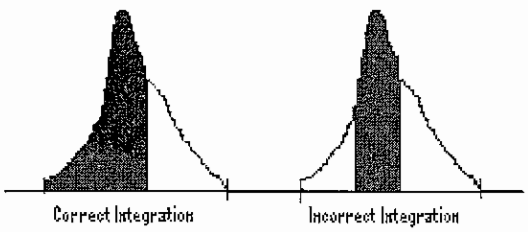


(059-014)

Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

(059-015)

Attachment 9.

List of Poor Purging or Poorly Performing Compounds

Acetone	Ethyl Acetate
Acetonitrile	2-Hexanone
Acrolein	Isobutyl Alcohol
Acrylonitrile	Methacrylonitrile
Bromomethane	Methyl Acetate
n-Butanol	4-Methyl-2-pentanone
2-Butanone (MEK)	2-Nitropropane
Carbon Disulfide	Propionitrile
Chloroethane	Tetrahydrafuran
2-Chloroethylvinyl ether	Trans-1,4-dichloro-2-butene
Chloromethane	1,1,2-Trichloro-1,2,2-trifluoromethane
Dichlorodifluoromethane	Trichlorofluoromethane
1,2-Dibromo-3-chloropropane	Vinyl Acetate

Attachment 10.

DOD QSM Version 4.2: Appendix F QC Requirements Summary (Table F-1; and Table F-4) (061-001 to 061-005)

Table F-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081: Endrin, DDT 8270: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of 'either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	All positive results must be confirmed.
Continuing calibration verification (CCV)	This verification of the ICAL that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL curve.	If the values for the analytes are outside the acceptance criteria, the ICAL may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analytical Capability	QC samples are analyzed in series to verify ability to produce data of acceptable precision and bias.	To verify the ability to produce data of acceptable precision and bias for a specific instrument type, matrix, method, and analyst.	The average recovery of the spikes and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples may not be conducted until this check is successful.
Duplicate Sample (replicate)	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte.	The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration.	Any sample associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported	A sample matrix, free from the analytes of interest, spiked with known amounts of analytes or a material containing known and verified amounts of analytes.	Used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Assesses the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the precision and bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
MS	A sample prepared by adding a know mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.	To assess the performance of the method as applied to a particular matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty

(061-001)

Table F-1 Cont.			
QC Check	**Definition**	**Purpose**	**Evaluation**
MSD	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of recovery for each analyte	To assess the performance of the method as applied to a particular matrix and provide information on the homogeneity of the matrix. Also used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. If the sample matrix is homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
MB	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with an under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure lab accuracy/bias. The sample could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or qualify (B-flag) all results for the specific analytes(s) in all samples in the associated prep batch as appropriate. For common lab contaminants, no analytes detected > RL. See Section D.1.1.1 and Box D-1
RT window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the RT window (i.e. start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the ICAL.	To idendify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
Second source calibration verification (ICV)	A standard obtained or prepared from a source independent of the source of standards for the ICAL. Its concentration should be at or near the middle of the calibration range. It is done after the ICAL.	To verify the accuracy of the ICAL.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared with the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer	Proper tuning of the mass spectrometer must be verified prior to sample analysis

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(061-002)

Table F-4: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method- specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD Determination and verification (See Box D-13)					
LOQ Establishment and verification (See Box D-14)					
Tuning	Prior to ICAL and at the beginning of each 12- hour period.	Refer to method specific ion criteria	Retune instrument and verify. Rerun affected samples	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check DDT (8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for DDT Benzidine & PCP should be present at their normal responses and should not exceed a tailing factor of 2.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<u>1. Average response factor (RF) for SPCCs:</u> VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 <u>2. RSD for RFs for CCCs:</u> VOCs and SVOCs ≤ 30% and one option below. **Option 1:** RSD for each analyte ≤ 15% **Option 2:** linear least squares regression: r ≥ 0.995 **Option 3:** non-linear regression-coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
2nd Source calibration verification (ICV)	Once after each ICAL	All project analytes within ± 20% of true value	Correct problem and verify 2nd source standard. Rerun, 2nd source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

(003-190)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
RT window position establishement for each analyte and surrogate	Once per ICAL	Position shall be set using midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Labs may update the RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the lab must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the RTs.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. %Differenee/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that failes, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards verification	Every field sample, standard, and QC sample.	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method Blank (MB)	One per prep batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no analytes detected > RL (see Box D-1).	Correct problem, then see criteria in box D-1; if required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

(400-190)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported, including surrogates	One per prep batch	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3 and Appendix G.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated prep batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per prep batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For specific analytes(s) in the parent sample, apply J-flag if acceptable criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate MSD or Sample Duplicate	One per prep batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client as to additional action measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for the failed surrogates in the associated prep batch, if sufficient sample material is available. If obvious chromatographic interferences with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interferences.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

Notes:
1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

(61205)

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COMPANY CONFIDENTIAL AND PROPRIETARY

TITLE: Sample Preparation Herbicides from a Waste Water Matrix or TCLP Extracts Using SW-846 8151A

Approvals (Signature/Date):

10/30/12	10/30/12
Daniel A. Knieriemen — Date Supervisor, Organic Extractions	Mark Storm — Date Env. Health & Safety Coordinator
10/30/12	10/30/12
Terese A. Preston — Date Quality Assurance Manager	Michael J. Healy — Date Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the extraction and cleanup procedures of chlorinated acid herbicides from a wastewater matrix using SW-846, 3rd Edition, Method 8151A as a reference.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable.

1.1.2 Reporting Limits

Not Applicable.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.3 Method Summary

Water samples and TCLP extracts are hydrolyzed using a solution of sodium hydroxide and extraneous material is removed by a DCM solvent wash. The samples are acidified with sulfuric acid and extracted with ethyl ether. The ethyl ether extracts are dried using acidified sodium sulfate and concentrated. The acids are converted to methyl esters using diazomethane and are determined by gas chromatography.

Because chlorinated phenoxy acid herbicides may occur in water in various forms, such as acids, salts, or esters, a hydrolysis step is included to permit the determination of the active part of the herbicide.

2.0 INTERFERENCES

- 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.
- 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 material.
- Soap residue on glassware may cause degradation of certain analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand-rinsed very carefully to avoid this problem. Because the herbicides react readily with alkaline substances, loss may occur if there is alkaline contact at any time except in the controlled alkaline hydrolysis step. The use of acid rinsed glassware and acidified sodium sulfate (Na_2SO_4) are used to help avoid this problem.

- Matrix interferences may be caused by contaminants that are co-extracted from the sample. These can appear as large, distinct peaks and/or elevated baselines. Occasionally matrix interferences may prevent the proper detection of surrogate and/or analytes, resulting in the reporting of low, or possibly high, surrogate and/or spike recoveries. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the site being sampled. Organic acids, especially chlorinated acids, cause the most direct interference. Phenols, including chlorophenols, may also interfere. Alkaline hydrolysis and subsequent extractions eliminate many of the predominant chlorinated pesticides.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The use of separatory funnels to extract aqueous samples with Methylene Chloride or Ethyl Ether creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.
- All glassware should be carefully inspected for any kind of crack or star prior to use. An imperfect separatory funnel could explode from the pressure that builds up when solvent is added. Dispose of imperfect glassware in an appropriate container. Some types of glassware are repairable.
- Take careful note of those sections marked **"CAUTION"** and **"NOTE"**.
- When pouring large volumes of solvents (100 mLs or greater), using a squeeze bottle, using the Cycletainer toggle, or transferring solvent in any manner such that a splash can occur, safety glasses are inadequate to protect the eyes and face. A face shield must be worn. A hood sash can act as a face shield when working with solvents inside a fume hood.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethanol	Flammable, Poison	1900 mg/m^3 per 10 hrs.	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Prolonged exposures to high concentration may cause drowsiness, loss of appetite, and inability to concentrate. If ingested will cause headaches, gastritis, intoxication, blindness and, in acute cases, death. Causes skin irritation, cracking or flaking due to dehydration and defatting action. Can cause eye irritation. Prolonged exposure may affect the nervous system, liver, kidneys, blood, g.i. tract and reproductive system. Continued ingestion of small amounts could result in blindness.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of with six months.**
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride (DCM)	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Glassware

- 2 Liter separatory funnels
- 100 mL, 1,000 mL graduated cylinder
- 1,000; 500 and 125 mL Erlenmeyer flasks
- 500 mL Kuderna-Danish flasks
- 3 Ball Snyder Columns
- 10 mL Receivers (insulated)
- Shallow Pyrex tray
- disposable transfer pipets
- Glass stirring rods
- 16 X 125 test tubes with Teflon lined lids
- Diazomethane generating apparatus

4.2 Miscellaneous

- Wide range pH paper 1 to 12
- Waterbath - heated, with concentric ring covers, capable of temp. control. Bath must be in a hood.
- Muffle Furnace
- Nitrogen Evaporator
- Rotary Evaporator
- Desiccator
- Centrifuge
- 100 uL microdispenser
- Boiling Chips, Teflon, soxhlet extracted
- Oven set at 130°C

5.0 REAGENTS AND STANDARDS

A label on any reagent bottle must contain the concentration of the reagent, name of the reagent, standard number, date prepared, expiration date and the analyst who prepared the reagent.

5.1 Reagents

5.1.1 Solvents

Acetone; Hexane; Methanol; Diethyl Ether; Methylene Chloride [a.k.a., Dichloromethane (DCM)] - Pesticide quality or better, meeting the requirements of the American Chemical Society Committee on Analytical Reagents. The Diethyl Ether must be free of peroxides as indicated by test strips.

5.1.2 Heat Purified Granular Sodium Sulfate

Sodium sulfate purchased directly from vendor. Purify by heating at 400°C for four hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year.
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

5.1.3 Heat-Purified Sodium Chloride

Sodium chloride purchased directly from vendor. Purify by heating at 400°C for four hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year.
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

5.1.4 Acidified Sodium Sulfate

Add ~1 mL concentrated H_2SO_4 to ~ 400 grams heat-purified sodium sulfate slurried with enough ethyl ether to just cover the solid. Decant the ethyl ether into an appropriate waste container. Place the "wet" sodium sulfate in a shallow drying pan, place in a shallow hot water bath, and allow the ethyl ether to evaporate (stirring often to avoid clumping). Mix 1g of resulting dry solid with 5 mL reagent water and confirm that the mixture has a pH < 4.

- Hazardous Properties: Sulfuric Acid (H_2SO_4) is extremely corrosive and toxic to tissues. Vapors are also harmful. See MSDS.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass container at 130°C.

5.1.5 Concentrated Sulfuric Acid (H_2SO_4)

Purchased from a vendor.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle

5.1.6 12 N Sulfuric Acid (H_2SO_4)

Cautiously add 333 mL concentrated sulfuric acid (~36 N), with mixing, to ~500 mL of reagent water. Bring up to 1 liter with reagent water. Mix thoroughly. Always make acid solutions in Pyrex glassware and a cool water bath due to the amount of heat given off by the reaction. **Always add acid to water.**

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle at 4 ± 2°C.

5.1.7 Sodium Hydroxide (NaOH)

Purchased from a vendor.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle

5.1.8 6 N Sodium Hydroxide Solution (NaOH)

Dissolve 240 g sodium hydroxide (NaOH) pellets into reagent water and dilute to 1.0 liter.

- Hazardous Properties: Toxic. Corrosive. Harmful if swallowed, inhaled or absorbed through skin. See MSDS for specifics.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle.

5.1.9 (1:9) Hydrochloric Acid (v:v) (HCl)

Add 100 mL of concentrated hydrochloric acid to 900 mL of reagent water.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle

5.1.10 Diazald

Purchased directly from a vendor.

- Storage Requirements: Store at 4±2°C.

5.1.11 Diazald Solution

Dissolve 5.0 grams diazald in 50 mL of ethyl ether.

- Hazardous Properties: Toxic, flammable. Harmful if swallowed, inhaled or absorbed through the skin. See specific MSDS for specifics.
- Life of Reagent: Prepare as needed

5.1.12 Potassium Hydroxide Solution (for esterification)

Dissolve 50 grams of KOH in 80 mLs of reagent water. Use 8 mL of this solution per batch of generated Diazomethane.

- Hazardous Properties: Toxic. Corrosive. Harmful if swallowed, inhaled or absorbed through skin. See MSDS for specifics.
- Life of Reagent: 1-year
- Storage Requirements: Store in a glass bottle.

5.1.13 (Trimethylsilyl)diazomethane, 2.0 M solution in hexanes

Purchased directly from a vendor.

- Hazardous Properties: Highly flammable, harmful if swallowed, inhaled or absorbed through skin.
- Life of Reagent: 1-year.
- Storage Requirements: Store in an amber glass bottle. Refrigerate after opening.

5.1.14 Silicic Acid, <20 micron, refined, 99.9%

Purchased directly from a vendor.

Hazardous Properties: Harmful if swallowed, inhaled or absorbed through skin.
Life of Reagent: 1 year
Storage Requirements: Store in an amber glass bottle.

5.1.15 Reagent Water

Defined as water in which no interferent is observed at one-half the reporting limit of any analyte when one liter of the reagent water is extracted and prepared by using the same procedure as for a water sample.

5.2 Quality Control (QC) Solutions

QC solutions must be stored in amber or foil-wrapped, glass containers that have Teflon-lined caps. The label on the container must contain the following information: Name of the solution, standard number, concentration of the components, date prepared, preparation analyst, and expiration date. All QC solutions are stored either in the standards refrigerator in the GC lab or in the standards refrigerator located in the organic extraction lab. QC solutions are stored at 4 ± 2 °C, in Teflon-sealed screw-cap bottles with minimal headspace and protected from light.

5.2.1 Surrogate Spike Solution (10X)

A concentrated solution of Dichlorophenyl Acetic Acid (DCAA) is purchased directly from a vendor. It is the responsibility of the GC/Extractables group to properly log-in these standards and to calculate expiration dates. The GC/Extractables group will prepare a solution of DCAA at ~50 ug/mL and label as a "Herbicide Surrogate Solution (10X)".

Compound	Concentration
DCAA	~50 ug/mL

- Life of Standard: Same expiration date as parent.
- Storage Requirements: 6 months from the date of preparation.

5.2.2 Spike Solution (10X)

A concentrated solution of Herbicide compounds is purchased directly from a vendor. It is the responsibility of the GC/Extractables group to properly log-in these standards and to calculate expiration dates. The concentration of the spike compounds are listed below.

Compound	Concentration ug/mL
Dalapon	57.0
4-Nitrophenol	47.0
Dicamba	9.8
Dichloroprop	52.0
2,4-D	46.0
Pentachlorophenol	6.0
2,4,5-TP	10.0
2,4,5-T	10.0
2,4-DB	54.5
Dinoseb	10.2
Picloram	6.0

- Life of Standard: Same expiration date as parent.
- Storage Requirements: 6 months from the date of preparation.

5.2.3 Surrogate Spike Solution Working

The surrogate used in the analysis of herbicides is Dichlorophenylacetic acid (DCAA). The GC/Extractables group prepares the surrogate solution. The desired concentration of the surrogate is listed below:

Compound	Concentration (in acetone)
DCAA	5 ug/mL

- Life of Standard: 6 months from the preparation date (or as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

5.2.4 Matrix Spike Solution Working

The GC/Extractables group prepares the matrix spike solution. The concentration of the spike compounds are listed below:

Compound	Concentration (ug/mL – in MeOH)
Dalapon	5.7
4-Nitrophenol	4.7
Dicamba	0.98
Dichloroprop	5.2
2,4-D	4.6
Pentachlorophenol	0.60
2,4,5-TP	1.0
2,4,5-T	1.0
2,4-DB	5.45
Dinoseb	1.02
Picloram	0.60

- Life of Standard: 6 months from the preparation date (or as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

6.0 CALIBRATION

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

The following QC is required with each organic extraction batch. An extraction batch may contain no more than 20 samples (excluding QC samples).

7.1.1 Method Blank (MB)

The MB is reagent water, which is spiked with the appropriate surrogate compound(s) (Sec. 5.2.1), taken through the entire extraction procedure to demonstrate that all materials used are interference-free; and is used to monitor the introduction of contaminants during the extraction process.

7.1.2 Laboratory Control Sample (LCS)

The LCS is reagent water which has been spiked with predetermined quantity of spike solution containing the designated herbicide compounds (Sec. 5.2.2).

7.1.3 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

A known concentration of matrix spike compounds, which are selected analytes, is added to aliquots of the sample matrix prior to extraction. These will be referred to as the MS/MSD.

7.1.4 Surrogates

Surrogates, compounds similar to the analytes in composition and chromatography, but not found in the environment, are added to each sample, MB, LCS and MS/MSD prior to extraction.

7.2 Sample Preservation and Storage

Sample container, preservation technique and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract/client requests. In general, samples collected for herbicide extraction from water are collected in 32 oz., 1 liter, or 80 oz. narrow mouth, amber, glass jars with Teflon-lined screw-cap lids. If a Teflon-lined lid is not available, solvent-rinsed aluminum foil can be used as a liner.

Samples, sample extracts and standards are stored separately.

NOTE: If the sample matrix is highly acidic or basic, corrosion of the foil may occur and contaminate the sample.
Samples are stored at 4 ± 2°C prior to extraction. Any samples received by the preparation group that do not comply must be noted. Deviations in results "could" be explained by the failure of sample collector / client / custodian to comply to sample collection and storage requirements.

Reference	Holding Time (vts)	Preservation
SW-846	7 days	Cool 4 ± 2°C

vts= verified time of sample collection

7.3 Sample Preparation

7.3.1 Glassware Preparation

All glassware (except Snyder columns, volumetric flasks, volumetric pipets, and syringes) must be washed thoroughly with warm soapy water, rinsed five times with hot tap water and rinsed three times with DI water. The stopcocks of the separatory funnels should be cleaned with a small brush. **It is absolutely mandatory that all glassware be scrupulously clean.** Failure to rinse well with water, may leave a soap film that is incompletely removed with solvent rinsing, and with subsequent extraction, "soap" peaks will appear in the chromatograms.

If glassware is very wet it may be towel dried. Glassware used for herbicide extractions must be rinsed with (1:9) hydrochloric acid. This step neutralizes any basic sites present on the glassware. **(Note: Glassware in which a base will be used, such as the separatory funnels used for the 2nd extraction, does not need to be rinsed with acid prior to use.)** Dispose of the acid in an appropriate waste vessel. **All** Glassware should be rinsed with cleaning grade acetone, and the excess should be discarded into the appropriate waste container. This step is done to ensure that the glassware is dry. **If after rinsing once, the glassware still contains any water, repeat the acetone rinse.** At this point let the glassware dry before continuing to ensure that all of the water is gone. All glassware should then be rinsed with ethyl ether using a Teflon squeeze bottle, and the excess discarded into the appropriate waste container. Repeat this step at least three times and allow the glassware to dry. Failure to remove all water prior to ethyl ether rinsing prevents the ethyl ether solvent from coming into contact with all surfaces on the glassware. The solvent rinse is then incomplete, and may leave contaminants on the glass, which are then co-extracted with the sample.

NOTE: Ethyl ether is considered a health hazard. It is harmful if swallowed or inhaled. The vapor is irritating and thus should be used in a hood. It is readily absorbed through the skin and contact may be irritating.

CAUTION: Pressure builds up quickly in a separatory funnel that has both the stopcock closed and the stopper in place. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Be sure to vent the separatory funnel frequently by inverting the funnel and opening the stopcock. Do not point the funnel at or near anyone as it is being vented. Always vent separatory funnels into a hood.

CAUTION: Do not use separatory funnels with stars or cracks, as these points may give under pressure.

7.3.2 Procedures for Return of Glassware to Dishroom

Any glassware that has organic residue present, all K-D receivers, and all separatory funnels should be rinsed with cleaning grade acetone, or whatever other solvent which removes the residue, prior to return to the dishroom from the lab. Glassware in which the residue can not be removed should be segregated from other glassware being returned to the dishroom. Glassware should be carefully placed in a tub and placed on the "Dirty" side of the dish racks. In order to reduce glassware breakage, glassware should not be stacked in the tubs. Severely chipped glassware should be removed from service. Some items are repairable. Check with your supervisor as to what items get repaired.

7.4 Calibration / Standardization

Not Applicable.

7.5 Preventive Maintenance

- Balance should be cleaned and checked for levelness prior to each use. Class "S" weights should be weighed and documented each day the balance is used. Consult your supervisor if the weights are out of acceptable limits. Do not use the balance until the situation is back in control.
- The K-D waterbath should contain DI water only. The water level should be checked prior to each use, and should not fall below the heating coils. If the heating coils do become exposed to the air while in use, they could burn-out and would require replacement. Keep the water level approximately one inch from the top of the bath edge.
- The N-Evap waterbath should also contain DI water which should not exceed 35°C. The water level in the bath should be sufficient to cover 3/4 of the receivers when submerged.
- The water level in the evaporation waterbath must be monitored so that it covers the drying pan adequately. Only DI water may be used in the waterbath. Set the temperature dial to 70°C. Monitor the temperature during the evaporation process.
- The thermometer used in the extraction waterbaths are checked against a reference NIST thermometer on a yearly basis or sooner if necessary.
- Periodically, the desiccant used in the desiccator should be removed and dried in an oven at 130°C.
- To avoid the breakage of sample extracts, the analyst needs to take care that the containers are properly balanced on the centrifuge. The container should fit snugly in the holder. Use a paper towel to cushion the centrifuge bottles if necessary.

7.6 Sample Analysis

7.6.1 Analytical (Preparation) Sequence

The following preparation sequence will be required of each extraction batch of at most, 20 samples:

Quality Control	Frequency
MB	1 per extraction batch of at most 20 samples
LCS [1]	1 set per extraction batch
MS /MSD [2]	At least 1 set in 20 samples (sample may be designated by the client)
TCLP Blanks	As determined by TCLP sets
Surrogate	Every sample / MB / LCS / MS / MSD

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.6.2 Sample Set-Up

7.6.2.1 Add 950 mL of reagent water to the separatory funnels to be used for the MB and LCS.

7.6.2.2 Prior to pouring water samples, sample containers should be inverted a few times to mix the sample. However, if there is sediment present, consult your supervisor or section manager before mixing. Also, if the sample appears to have more than one phase, immediately contact your supervisor, section manager or project manager. The client will need to be notified.

NOTE: If the sample container is of a 1-liter or 1-quart size (**and the entire sample is being used**), the first aliquot of extraction solvent should be used to rinse the sample container.

7.6.2.3 The sample volume used for waste waters and TCLP extracts is different. For waste waters, pour the entire sample into a 1,000 mL graduated cylinder and record the volume. Transfer the water samples from the 1,000 mL cylinder to a labeled, 2 liter separatory funnel. Measure out two additional aliquots (three total) of the sample chosen/designated to have a MS/MSD performed on it (use 950 mL aliquots for MS/MSD if sample volume allows). For TCLP extracts, measure 10 mLs of each sample (including the TCLP blanks) and pour into individually labeled separatory funnels that already contain 900 mLs reagent water. Measure another 10 mL aliquot of each sample that requires a matrix spike and pour into another separatory funnel that already contains 900 mLs reagent water.

* Alternatively, pour the entire sample into a 1,000 mL Erlenmeyer and **mark the meniscus**. The volume will be measured exactly at the end of the extraction by refilling to the marked line and transferring to a graduated cylinder to measure volume.

7.6.2.4 Take an initial pH by dipping a transfer pipet into the sample and touching a drop on wide range pH paper. Record the initial pH in the extraction worksheet.
NOTE: All spiking solutions MUST be at room temperature before use. This includes stock solutions when preparing working spike solutions.

Actual concentrations will change at colder temperatures. Furthermore, some components may come out of solution in the freezer. Warming to room temperature reverses the process.

7.6.2.5 Waste waters and TCLP extracts use the same surrogate standard. For waste waters, add 1.0 mL of the surrogate working solution (Sec. 5.2.3) to every MB, LCS, sample, and MS/MSD.

7.6.2.6 Waste waters and TCLP extracts use the same matrix spike standards. For waste waters, add 1.0 mL of the matrix spike working solution (Sec. 5.2.4) to the LCS and MS/MSD.

7.6.2.7 Add 250 g of heat purified sodium chloride to each sample, seal, and shake to dissolve the salt.

7.6.3 Hydrolysis

7.6.3.1 Add 17 mL of 6 N NaOH to the sample and swirl vigorously. Check the pH of the samples with pH paper. If the pH is not $\geq$ 12, add more 6 N NaOH.

7.6.3.2 Let the sample sit at room temperature 1-2 hours, shaking the separatory funnel periodically.

7.6.4 Basic Solvent Wash

Add 60 mL of DCM to the samples. Extract the samples by shaking vigorously for 2 minutes, with periodic venting to release excess pressure. 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 one or more of the following techniques to complete the phase separation. Document the presence of severe emulsions in the log book.

7.6.4.1 Stirring Rod

Physically break up the emulsion by stirring.

7.6.4.2 Centrifugation

Centrifugation separates by density. Draw off the emulsion into a 125 mL, Teflon, centrifuge bottle. Balance the centrifuge. Centrifuge 2-5 minutes. Carefully pour the contents back into the separatory funnel and drain off the DCM layer. Alternatively, after centrifugation, draw out the DCM (bottom layer) directly from the centrifuge bottle using a disposable, 10 mL pipet and discard it. Return any aqueous phase back to the appropriate separatory funnel. Add the next DCM aliquot first to the centrifuge bottle as a rinse and then to the appropriate separatory funnel.

7.6.4.2.1 Drain and discard the DCM layer (bottom layer).

7.6.4.2.2 If necessary perform a second and third DCM wash in the same manner.

7.6.5 Acid Extraction

7.6.5.1 Add 17 mL of cold 12 N sulfuric acid to the samples. Swirl vigorously and check the pH. If the pH is not ≤ 2, add more 12 N sulfuric acid. Recheck the pH.

7.6.5.2 Add 120 mLs of ethyl ether to each sample contained in the separatory funnel. Shake the separatory funnel vigorously for 2 minutes. 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 one or more of the following techniques to complete the phase separation. Document the presence of severe emulsions in the extraction batch information sheet.

7.6.5.3 Stirring Rod

Physically break up the emulsion by stirring.

7.6.5.4 Centrifugation

Centrifugation separates by density. Draw off the emulsion into a 125 mL, Teflon, centrifuge bottle. Balance the centrifuge. Centrifuge 2-5 minutes. Carefully pour the contents back into the separatory funnel and continue with 7.6.6. Alternatively, after centrifugation, draw out the ethyl ether (top layer) directly from the centrifuge bottle using a disposable, 10 mL pipet and place in the appropriate collection flask. Return any aqueous phase back to the appropriate separatory funnel. Add the next ethyl ether aliquot first to the centrifuge bottle as a rinse and then to the appropriate separatory funnel.

7.6.6 Drain the aqueous layer (lower layer) into the 1,000 mL Erlenmeyer flask. Drain the ether solvent layer (top layer) into a 500 mL Erlenmeyer flask. Pour the aqueous sample back into the separatory funnel.

7.6.7 Repeat the extraction process twice more using fresh 60 mL aliquots of ethyl ether.

CAUTION: Pressure builds up quickly in a separatory funnel that has both the stopcock closed and the stopper in place. Initial venting should be done immediately after the separatory funnel has been sealed and shaken once. Be sure to vent the separatory funnel frequently by inverting the funnel and opening the stopcock. Do not point the funnel at or near anyone as it is being vented.

7.6.8 Sample Concentration

7.6.8.1 Add ~5-10 grams of acidified sodium sulfate (Sec. 5.1.3) to each ethyl ether sample extract. Swirl the flask and verify that sufficient acidified sodium sulfate has been added. **

** **NOTE:** The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free-flowing crystals are visible when swirling the flask. If all the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling.

The extract must stay in contact with the acidified sodium sulfate for a minimum of 2 hours. If the extracts are not going to be concentrated right away, do not add the acidified sodium sulfate until 2-3 hours before concentrating.

7.6.8.2 Assemble a K-D concentrator by attaching a 10 mL, **insulated**, concentrator tube to a 500 mL K-D flask. Add a couple of boiling chips. Transfer each extract to its own K-D concentrator (making sure that each flask is appropriately labeled). Rinse the Erlenmeyer flask 3 times with ethyl ether, adding each rinse to the K-D flask. Rinse down the top joint of the K-D flask with ethyl ether and attach a 3-ball Snyder column. Be careful not to allow any sodium sulfate crystal to be transferred to the K-D apparatus.

7.6.8.3 Place each K-D flask on the hot waterbath. Set the temperature control to either low or medium in order to achieve a temperature between 60-70° C. The balls of the Snyder column should chatter but the chambers should not flood.

7.6.8.4 When the apparent volume reaches about 2-5 mL, remove the K-D flask from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs ethyl ether. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove any water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint into the receiver with a little ethyl ether. Cover the receiver with a piece of aluminum foil. Quantitatively transfer each sample to an appropriately labeled screw top test tube that has been previously marked at 10 mL.*

***NOTE:** Volumetrically pipet 10 mL of ethyl ether into a 16 X 125 test tube. Mark the meniscus, using a fine-tipped marker. Discard the ethyl ether into an appropriate waste vessel.

7.6.9 Nitrogen Evaporation

7.6.9.1 For further concentration, place each receiver or test tube on the N-Evap tray. The N-Evap outlet tubes should be stored in a 600 mL beaker containing DCM:Acetone (1:1) mixture prior to each use. Wipe each outlet tube with a KimWipe and lock into place on the N-Evap. Lower the outlet tube into the receiver being careful not to touch the sides of the receiver. The outlet tube should remain above the surface of the extract. Open the valve of the nitrogen tank and adjust the pressure in order to achieve the appropriate nitrogen stream. The nitrogen stream should make a slight "dimple" in the surface of the extract. If the extract bubbles, the nitrogen stream is **not** gentle enough. Lower the entire N-Evap tray into the warm water bath to aid the evaporation process. Heat is not necessary and if used the temperature should not exceed 35°C.

7.6.9.2 The extracts must never be allowed to go dry. While the extracts are concentrating, occasionally rinse down the sides of the receiver with small aliquots of ethyl ether.

7.6.9.3 The final extract volume is dependent upon the process used for esterification. For the (Trimethylsilyl)diazomethane procedure (Section 7.6.10.1), concentrate the sample to ~2 mL and add 100 uL of methanol to each extract. For the Diazomethane generation from Diazald procedure (Section 7.6.10.2), dilute the extract with 1 mL isooctane and 0.5 mL of methanol and bring the extract up to a final volume of 4 mLs with ethyl ether.

7.6.9.4 To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC extractables lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the GC extractables group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

7.6.10 Esterification

There are two different esterification methods which may be used; Diazomethane generation from Diazald, and the (Trimethylsilyl)diazomethane procedure. The (Trimethylsilyl)diazomethane procedure is preferred. However, if circumstances require, the Diazald generation procedure may be employed.

Both methods must be performed under a hood.

7.6.10.1 (Trimethylsilyl)diazomethane procedure

7.6.10.1.1 Add the (Trimethylsilyl)diazomethane solution drop wise to each sample test tube (sample extract is at ~2 mLs) until the extract then turns a yellow color and the color persists. During the esterification reaction the extract will bubble. Continue to add the solution until the bubbling no longer occurs.

7.6.10.1.2 After one hour the reaction is halted and the diazomethane is removed by the addition of ~ 100 mg of silicic acid. Interferences will occur if too much silicic acid is added.

7.6.10.1.3 The extract is then brought up to a final volume of 10.0 mLs with hexane.

7.6.10.2 Diazomethane generation from Diazald

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

- Do not heat above 90°C---Explosion may result
- Avoid grinding surfaces, ground glass joints---Explosion may result
- Store away from alkali metals---Explosion may result
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride and boiling chips

7.6.10.2.1 The Diazald generation apparatus should be pre-assembled under an extraction hood in the extraction room. Warm the extraction vessel to 65°C. Add water if necessary. Add 10 mL of 95% ethanol to 6 mL of potassium hydroxide solution [50 g KOH in 800 mL water] in a reaction vessel. Add a solution of Diazald [(5.0 g) in 50 mL of ether] to the separatory funnel (stopcock closed). Fill the bath around the collection flask with a mixture of 33 g sodium chloride and 100 g of crushed ice. Have more ice ready if necessary. **Once the temperature of the reaction vessel bath reaches 65°C, cautiously add the diazald ether solution to the reaction vessel over a period of 20 minutes.** The rate of addition should approximate the rate of distillation. Add more ice if necessary. When all the Diazald has been used up, slowly add 10 mL of ether and continue the distillation until the distillate is colorless.

7.6.10.2.2 Add 2 mL of the diazomethane distillate as prepared above to each sample test tube (sample extract at ~ 2 mL -- mark the meniscus). Let the sample/diazomethane stand for 20 minutes, occasionally swirling. Place the test tube on the nitrogen evaporator (See Sec. 7.6.9) and evaporate off the diazomethane (the yellow color from the diazomethane will disappear).

7.6.10.2.3 Add 2 mL of hexane to each test tube. Concentrate the extracts back down to the 2 mL mark (** see section 7.6.9). Using hexane, adjust the final volume of each extract to the 10.0 mL mark.

7.6.10.2.4 Store extracts at 4 ± 2° C prior to analysis.

7.6.11 Screening and Cleanup

Samples are not pre-screened prior to extraction.

Sludge samples are a mixture of water and suspended solid materials. If the percent moisture of a given sludge is greater than 90%, a 10 g or 30 g portion may be weighed and added to 1,000 mL of reagent water. It is then extracted like a water matrix as described in this procedure. Aqueous drum liquids may be prepared in the same manner. Despite the extraction method, sludges are generally reported on a dry weight basis and the total solids result must be entered into the spreadsheet.

At least one base cleanup is performed on all water extracts.

7.7 Documentation

All extraction information must be carefully documented in 'real time' in the L batch and prep worksheet (Attachment 1). Any additional sample or extraction information is recorded in the comment section of the batch sheet. This includes any additional sample or extraction information; and problems and/or deviations from normal procedure. The supervisor should be notified and a Non-Conformance Memo (NCM) may need to be initiated.

An extraction batch contains samples of similar matrix that are logged in for the same method code.

An extraction batch may contain different analyte list identifiers.

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 At least one MB and LCS will be included in each extraction batch of not more than 20 samples. The MBs will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine both precision and accuracy.

8.1.2 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable.

8.1.3 Precision will be measured by the reproducibility of the LCS between batches and will be calculated as Relative Percent Difference (RPD). Results must agree within statistical control limits in order to be considered acceptable.

8.1.4 The MS and MSD monitor the effect of sample matrix on method precision and accuracy, but are not used to determine if reanalysis is required.

8.1.5 Surrogate compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable.

8.2 Corrective Action

8.2.1 The extraction analyst must perform the Quality Control described in Section 9.2. The extraction analyst will not know if all QC is in control until the GC group analyzes the extracts and determine the results of the MBs, LCSs, MS/MSD and surrogate recoveries. It is the responsibility of the GC group to inform the Organic Extractions Department, by initiating a Non-Conformance Memo (NCM) in LIMs, when a sample or set of samples requires re-extraction. Re-extractions are to be designated as R1 in the extraction process.

8.2.2 All problems and/or deviations from normal procedures must be documented in the extraction logbook. The supervisor should be notified and a SDR may need to be initiated.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste flammable solvents. Solvents will be collected in a waste jar and poured into the Waste Chlorinated Solvent drum using a funnel to reduce splashing.
- Extracted water samples will be poured into the Waste Water drum using a funnel to reduce splashing.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1. Example: Extraction Prep Worksheet.
Attachment 2: Flowcharts for Herbicide Extraction and Herbicide Esterification

14.0 REVISION HISTORY

- Revision 11, was updated on 10/30/12
- Annual Review
- Section 7.6.8. Delete reference to Sample Concentration Log.

Attachment 1.

Example: Extraction Prep Worksheet (021-001 to 021-004)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-167901 | Analyst: Walker, Natalie M | Batch Open: 10/30/2012 5:21:51PM

Method Code: 500-8151A_AP-500 | Batch End: 10/30/2012 8:00:00PM

Extraction (Herbicides)

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	GrossWt TareWt	InitAmnt FinAmnt	Rcvd	PHs Adj1	Adj2	Due Date	Analytical TAT	Dlv Rank	Comments
1	MB 500-167901/1	MB~500-167901/1 N/A	N/A		1000 mL	6	12	2	N/A	N/A	N/A	
					10.0 mL							
2	LCS 500-167901/2	LCS~500-167901/2 N/A	N/A		1000 mL	6	12	2	N/A	N/A	N/A	
					10.0 mL							
3	LB 500-167718/1-A	LB~500-167718/1-A N/A	N/A		10.0 mL	6	12	2	N/A	N/A	N/A	
					10.0 mL							
4	500-51721-A-2-C	500-51721-A-2-C (8151A)	N/A		10.0 mL	6	12	2	10/31/12	4_Days - R	4	
					10.0 mL							
5	500-51721-A-2-C MS	500-51721-A-2-C~MS (8151A)	N/A		10.0 mL	6	12	2	10/31/12	4_Days - R	4	
					10.0 mL							
6	500-51721-A-5-B	500-51721-A-5-B (8151A)	N/A		10.0 mL	6	12	2	10/31/12	4_Days - R	4	
					10.0 mL							

(100-120)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-167901 | Analyst: Walker, Natalie M | Batch Open: 10/30/2012 5:21:51PM

Method Code: 500-8151A_AP-500 | Batch End: 10/30/2012 8:00:00PM

Batch Notes

Diethyl Ether Lot #	1976508, 1976520
Sulfuric Acid Lot Number	1938765
Hexane Lot#	1969742
MeCL2 Lot #	1957234
MTBE Lot #	
Na2SO4, Acidified Lot #	1950077
NaCL Lot #	1971337
NaOH Lot #	1954446
TMSDM Lot #	1962865
Concentration Start Time	1730
Concentration End Time	1900
Water Bath Temperature	
Uncorrected Temperature	
MeOH, Acidified Lot #	
Potassium Hydroxide Lot #	
Carbitol Lot #	
Diazald Lot #	
Silicic Acid Lot #	1440229
Silica Gel Lot Number	
N-evap #	C-0655
N-evap temperature	29.9

(021-002)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-167901 | Analyst: Walker, Natalie M | Batch Open: 10/30/2012 5:21:51PM

Method Code: 500-8151A_AP-500 | Batch End: 10/30/2012 8:00:00PM

Uncorrected N-evap Temperature	30.0
Sufficient volume for MS/MSD?	Y
Pipette ID	
Syringe Lot #	B18, B22
Batch Comment	

Comments

(021-003)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-167901 | Analyst: Walker, Natalie M | Batch Open: 10/30/2012 5:21:51PM

Method Code: 500-8151A_AP-500 | Batch End: 10/30/2012 8:00:00PM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
MB 500-167901/1	EXHERBSUW_00133	1000 uL	10.0 mL		
LCS 500-167901/2	EXHERBSUW_00133	1000 uL	10.0 mL		
LCS 500-167901/2	HESP1X_00027	1000 uL	10.0 mL		
LB 500-167718/1-A	EXHERBSUW_00133	1000 uL	10.0 mL		
500-51721-A-2-C	EXHERBSUW_00133	1000 uL	10.0 mL		
500-51721-A-2-C MS	EXHERBSUW_00133	1000 uL	10.0 mL		
500-51721-A-2-C MS	HESP1X_00027	1000 uL	10.0 mL		
500-51721-A-5-B	EXHERBSUW_00133	1000 uL	10.0 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

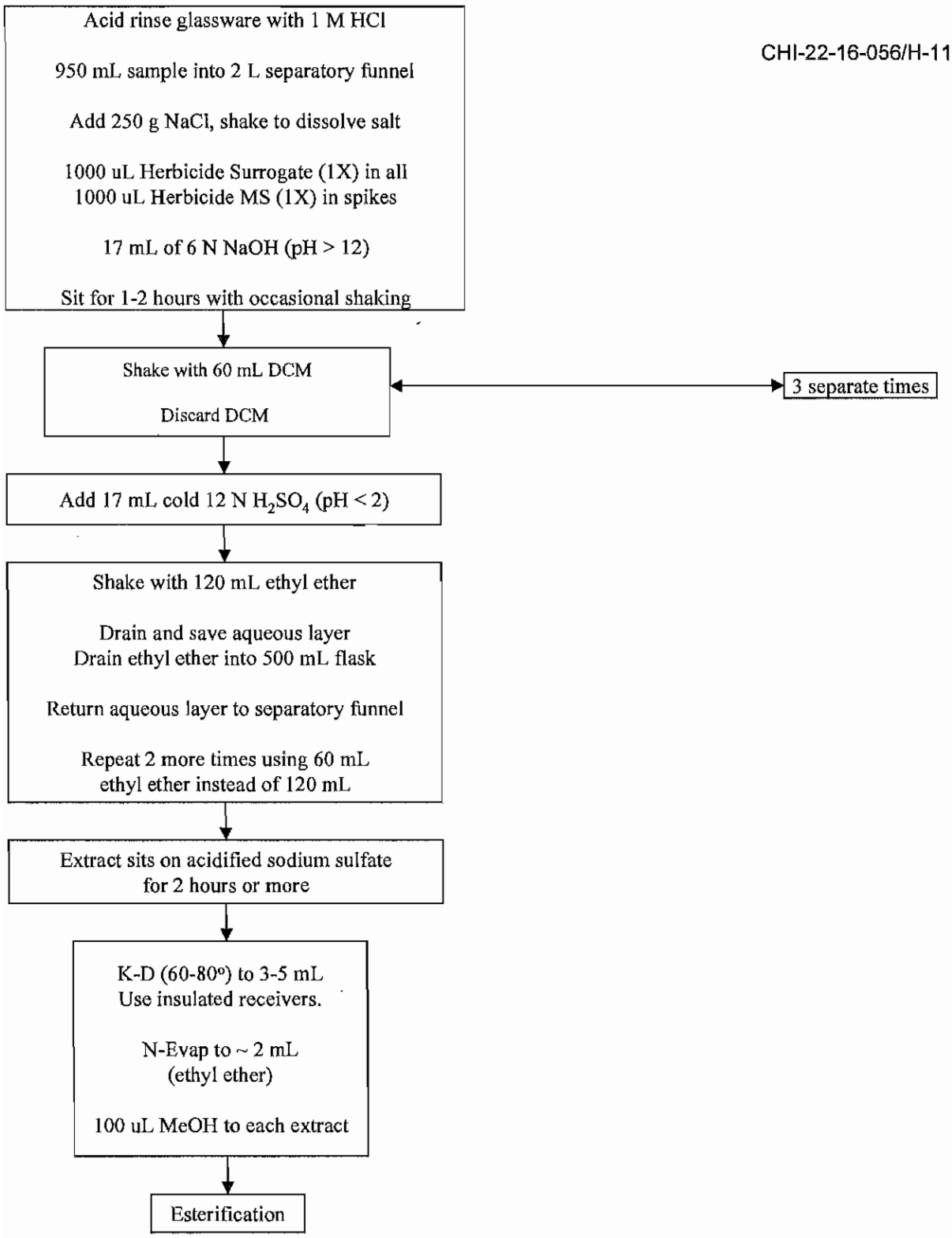
(021-004)

Attachment 2.

Example: Flowcharts for Herbicide Extraction and Herbicide Esterification (022-001 to 022-003)

Flowchart for Herbicide Waters (8151A)
8151A_AP

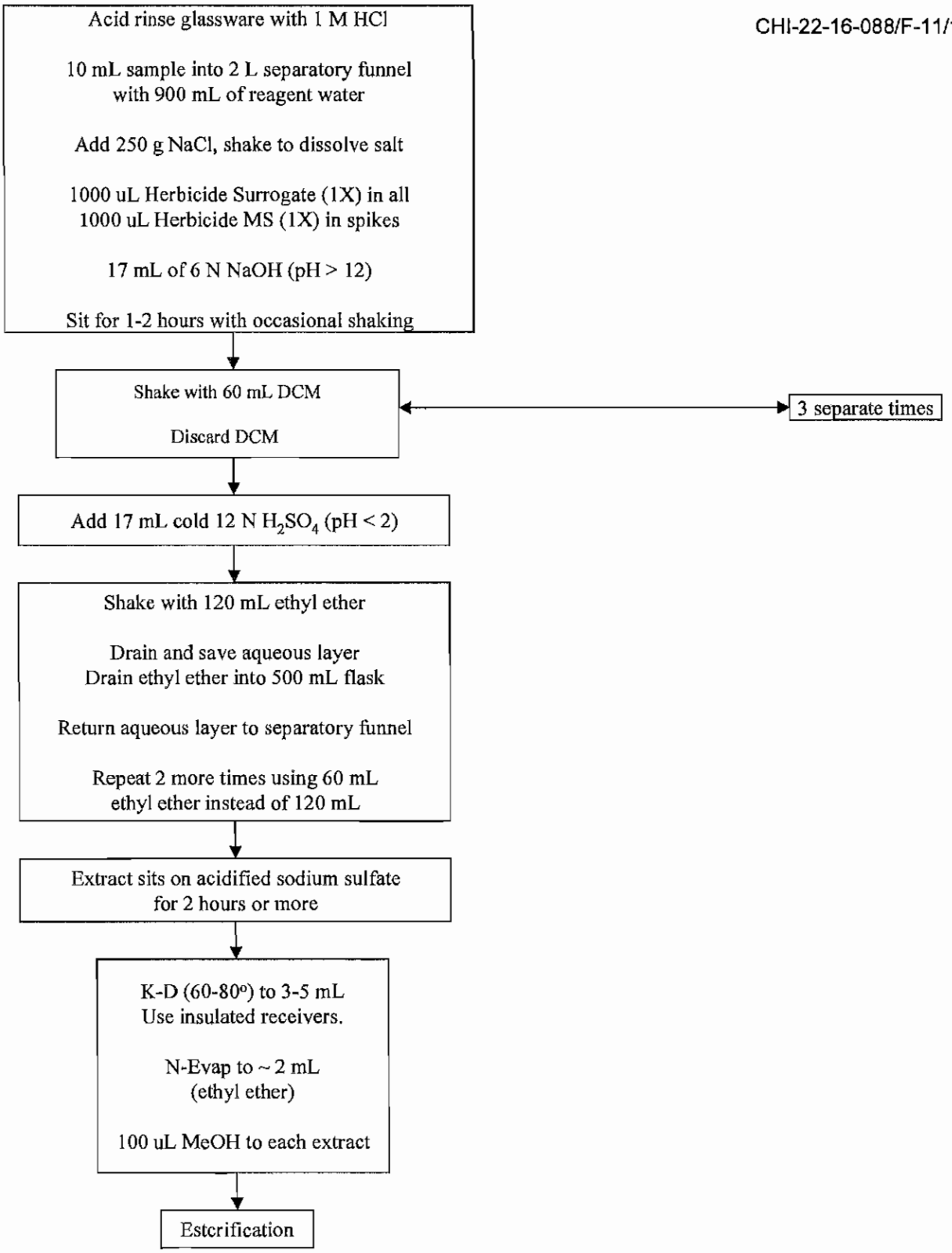
CHI-22-16-056/H-11/12



(022-001)

Flowchart for Herbicide TCLP (8151)
8151A_AP

CHI-22-16-088/F-11/12



(0 22-002)

Flowchart for Herbicide Esterification

(Trimethylsily)diazomethane Method

Exchange extract to ~ 2 mLs hexane.

↓

Add (Trimethylsily)diazomethane solution drop wise to each test tube until the extract turns yellow and color persists and bubbling stops.

Let stand for one hour

↓

Add ~ 100 mg of silicic acid to halt reaction

Bring up to 10.0 mLs in hexane

↓

Relinquish

CHI-22-16-089/H-11/12

Diazomethane generation from Diazald

Warm reaction vessel bath to 65°C

Add 10 mL 95% ethanol to 6 mL KOH solution in reaction vessel

Add Diazald solution to addition funnel (5.0 g in 50 mL ethyl ether)

Fill collection flask bath with 33 g of NaCl and 100 g of crushed ice

↓

When reaction vessel bath reaches 65°C add Diazald ether solution over 20 min

↓

When Diazald is completely transferred slowly add 10 mL ethyl ether

Continue distillation until Diazald is completely consumed (distillate is colorless)

↓

Add 2 mL diazomethane distillate

Let stand for 20 min

Evaporate off remaining diazomethane on N-Evap (yellow color will disappear)

Add 2 mL hexane and N-Evap to 2 mL

Bring up to 10.0 mL in hexane

↓

Relinquish

(022-003)

TestAmerica Chicago
STANDARD OPERATING PRACTICE (SOP) CHANGE FORM

Original SOP Number/Revision #: UP-SP-1311, Rev. 18 Last Mod ID (circle): NA/ _____

SOP Title: **SAMPLE PREPARATION: Toxicity Characteristic Leaching Procedure (TCLP)**

Affected SOP Section Number(s): 7.6.1.12
Effective Date of Change: 08/23/13

CONTROLLED DISTRIBUTION

COPY # :
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Full Signature Approvals Are Kept on File with TestAmerica Chicago Standard Practice Records

Revision Number with Mod ID: UP-SP-1311, Rv 18A

The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.**

1. Reason for SOP Change:

   ESI BP LaMP Audit Response.

2. Summary of Procedure Change (circle to indicate if there are attachments to this form: No / Yes: # pages attached = ___)

The following text is to be updated in Section 7.6.1.12:

- Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, using the example 1 mm grid as a visual reference.
- Transfer a 5.0 g portion to a 250 mL beaker recording the sample weight on the sample container and on the TCLP Backlog. The pre-test sample weight will be transferred to the TCLP pH Measurement Logbook for samples that need to follow the BP LaMP protocol and upon client request.

Diane L. Harper 8/22/13
Initiated/Reviewed By: Name/Date

Initiated/Reviewed By: Name/Date

Julie Johnson 8/22/13
Approval Signature/Date: Section Manager

Teresa A. Preston 8/22/13
Approval Signature/Date: QA Manager or Designee

TITLE: SAMPLE PREPARATION
Toxicity Characteristic Leaching Procedure (TCLP)

Approvals (Signature/Date):

Diane L. Harper 2/6/13 Diane L. Harper — Date Inorganics Manager	Debbie Johnson 2/6/13 Debbie Johnson — Date Supervisor, Metals Dept.
Mark A. Storm 2/6/13 Mark Storm — Date Env. Health & Safety Coordinator	
Terese A. Preston 2/6/13 Terese A. Preston — Date Quality Assurance Manager	Michael J. Healy 2/6/13 Michael J. Healy — Date Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the Toxicity Characteristic Leaching Procedure (TCLP). This SOP was written using 40 CFR 261 (Appendix II) and SW-846, 3rd Edition, Method 1311 as reference.

TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solids and multi-phasic wastes.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

Two distinct methods are utilized depending on whether volatile organics or other organic and metal constituents will be analyzed. A special zero-headspace extractor (ZHE) is used for volatile sample preparation and 2.0-Liter HDPE plastic or Teflon bottles are used for the other constituents.

- For solid wastes or wastes that contain significant amounts of solid material, the particle size is reduced and the liquid phase (if any) is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid that is equal to 20 times the weight of the solid material.

- A portion of the extract for metals analysis <u>only</u> is spiked by the TCLP analyst with the analytes of concern (at the regulatory level) and acidified with nitric acid to a pH < 2 (refer to Attachment 1). Note: If elements not contained in the TCLP spiking solutions are requested, the sample will be spiked (with the appropriate solution) by the metals prep analyst.

The TCLP sample is then analyzed by the appropriate method for organic and inorganic constituents. Refer to Figure 1 for the TCLP Flowchart; Table 1 for a listing of the Toxicity Characteristic Constituents and Regulatory Levels; and Table 2 for the maximum sample holding times.

2.0 INTERFERENCES

- Since this is a preparation procedure, interferences will only become apparent at the spiking and analysis stage. Interferences for spiking and for instrumentation are discussed in the analytical SOPs.
- A physical interference may occur for pH readings if the waste material is high in organic material (such as an oil). The waste may coat the pH probe, which affects the ability to obtain an accurate reading. When this type of interference occurs, pH paper is used instead of a meter for the final pH measurement. The use of pH paper is documented in the laboratory logbook.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The standards contain potentially harmful elements. Care should be taken to avoid contact with the stock solutions. In case of contact, rinse with cold water for 15 minutes.
- If contact occurs with a standard containing Hydrofluoric Acid, flush with water and apply Calcium Gluconate Gel (located in standards cabinet) immediately. Seek medical attention.

4.0 EQUIPMENT AND SUPPLIES

- The extractor is a custom made rotary-type design that meets the specifications of tumbling the samples at a rate of 30±2 RPMs, which is checked monthly and documented in the extraction logbook.
- 2-Liter plastic bottles (HDPE for inorganics).
- 2-liter Teflon bottles [For organics (BNA, Herb/Pest)].
- pH meter and paper - pH meter accurate to ±0.05 pH units at 25°C. Refer to Appendix 3 for details on meter calibration.
- Filtering apparatus - pressure filter using compressed Nitrogen as the purge gas.
- Zero Headspace extraction vessel (ZHE) - purchased unit for volatiles.
- 9.5 mm Sieve.
- Filter paper - glass fiber, 0.7 um pore size.

NOTE: Filters shall be made of borosilicate glass fiber. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1 N nitric acid followed by 3 consecutive rinses with deionized (DI) water (a minimum of 1 L per rinse is recommended).

NOTE: After a single use, Teflon tumbler jars and ZHEs shall be completely disassembled and all the parts washed in hot soapy water, followed by 3 rinses in tap water and three rinses in DI water, prior to re-assembly for the next sample. If there has been contamination with an oily substance, a solvent rinse may be necessary, followed by oven drying (ZHEs only) to evaporate the solvent.

- Lab balance capable of reading ± 0.01 g
- Tedlar Bags®
- ZHE Extraction Fluid Transfer Device - any device capable of transferring the extraction fluid to the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or a peristaltic pump, a gas tight syringe, pressure filtration unit).
- 40 mL VOA Vials
- Non-Coring stainless steel needle w/female luer lock. Male stainless steel luer lock adapter, non-coring stainless steel vent tube.

5.0 REAGENTS AND STANDARDS

All purchased acids, reagents, and dry chemicals used in this protocol must be ACS Reagent grade or better.

5.1 Hydrochloric Acid (HCl), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 83 mLs of concentrated hydrochloric acid. Swirl the flask to mix. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.2 Nitric Acid (HNO_3), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 64 mLs of concentrated nitric acid. Swirl the flask to mix. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.3 Sodium Hydroxide (NaOH), 1.0 N

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, add 40.0 g of sodium hydroxide pellets. Swirl the flask to mix. This is an **EXOTHERMIC** reaction. The flask should be placed in a cool water bath when mixing. Dilute to volume with DI water.

- Life of Reagent: 1 year
- Storage Requirements: None

5.4 Glacial Acetic Acid, Reagent Grade

Purchased.

- Life of Reagent: 1 year
- Storage Requirements: None

5.5 Extraction Fluid #1

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 5.7 mLs of glacial acetic acid. Swirl the flask to mix. Then add 64.3 mLs of 1.0 N sodium hydroxide solution (Rgt. 5.3) and swirl to mix once again. Dilute to volume with DI water. The pH of this extraction fluid should be 4.93 ± 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy, a class A graduated cylinder for adding the NaOH, and a re-pipettor to dispense the proportional amount of acetic acid.

- Life of Reagent: 1 day
- Storage Requirements: None

5.6 Extraction Fluid #2

To a 1-L Class A volumetric flask containing ~500 mLs of DI water, carefully add 5.7 mL of glacial acetic acid. Swirl the flask to mix. Dilute to volume with DI water. The pH of this Extraction Fluid should be 2.88 ± 0.05.

Note: For convenience, TestAmerica Chicago typically prepares this fluid in 20 liter batches, using a 20 liter carboy and a re-pipettor to dispense the proportional amount of acetic acid. Any volume can be prepared as long as the components are added proportionally and the pH is acceptable.

- Life of Reagent: 1 day
- Storage Requirements: None

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Refer to Section 8.1.

7.2 Sample Preservation and Storage

Parameter	**From:** Field Collection **To:** TCLP Extraction	**From:** TCLP Extraction **To:** Preparative Extraction	**From:** Preparative Extraction **To:** Determinative Analysis	**Total Elapsed Time**
Volatiles	14 days	NA	14 days	28 days
Semi-Volatiles [1]	14 days	7 days	40 days	61 days
Mercury	28 days	NA	28 days	56 days
Metals (except Hg)	180 days	NA	180 days	360 days

[1] BNAs, Pesticides and Herbicides
NA = Not Applicable

7.3 Sample Preparation / Size

7.3.1 Inorganics & Semi-Volatiles (BNAs, Pesticides and Herbicides)

Type of Sample	Sample Size
Samples containing 100% solids	100g solid
Samples containing 0.5% - 99.9% solids	100 g solid ideally, 75.0 g solid minimum
Samples containing < 0.5% solids	Refer to Section 7.6.1.14

7.3.2 Volatiles (ZHE)

Type of Sample	Sample Size
Samples containing 100% solids	25 g solid
Samples containing 0.5% - 99.9% solids	25 g solid
Samples containing <0.5% solids	Refer to Section 7.6.2.7

7.4 Calibration / Standardization

Refer to Attachment 3 for instructions on calibrating the pH meter.

7.5 Preventive Maintenance

- The main preventive maintenance required is keeping the area and all equipment clean and free of contaminants.
- The pH probe should be checked periodically for bubbles. The probes are replaced when needed.
- The ZHE's shall be checked for leaks after every use. When a malfunctioning ZHE is discovered, that particular ZHE or part will be identified with an 'Out-of-Service' tag or equivalent and removed from service.

7.6 Sample Extraction

7.6.1 Procedure when Volatiles are Not Involved

Although a minimum sample size of 100 grams is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough waste sample should be collected such that at least 75 grams of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (semivolatiles, metals, pesticides and herbicides).

The determination of which extraction fluid to use (Sec. 7.6.1.12) may also be conducted at the start of this procedure. This determination shall be on the solid phase of the waste (as obtained using glass fiber filter filtration).

7.6.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 100.0 g portion of the sample and proceed to 7.6.1.11.

7.6.1.2 If the sample is liquid that contains any visible sediment, even if logged in as a water sample, or if it is multi-phasic, liquid/solid separation is required. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water sample after a documented discussion with the laboratory.

This involves the filtration device outlined in Sections 7.6.1.3 through 7.6.1.9.

7.6.1.3 Pre-weigh the filter and the container which will receive the filtrate.

7.6.1.4 Assemble the filter holder and filter.

7.6.1.5 Weigh out a representative 100 g sub-sample of the waste and record the weight.

7.6.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.6.1.7 Transfer the waste sample to the filter holder.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.1.5 to determine the weight of the waste sample which will be filtered.

Gradually apply pressure of 10 psi, until gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any two minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter in any two minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, filtration is stopped.

7.6.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes will obviously contain some material that appears to be a liquid - but even after applying pressure filtration this material may not filter. In this case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

7.6.1.9 Determine the weight of the liquid phase by subtracting the total weight of the filtrate container (Sec. 7.6.1.3) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed (Sec. 7.6.1.15) or stored at 4 $\pm$ 2°C until it is checked for compatibility with the rotated extract (Sec. 7.6.1.15).

The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Sec. 7.6.1.5 or 7.6.1.7. Record the weight of the liquid and solid phases.

NOTE: If the weight of the solid phase of the waste is <75 g. Review the beginning of Section 7.3 about sample sizes.

7.6.1.10 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% solids. If the sample obviously has >0.5% solids, go to Sec. 7.6.1.11. If the sample is a liquid and contains any visible sediment, even if it is logged in as a water sample, the percent solids will be determined as follows. The only exception will be when a client has specifically agreed that the sample is to be analyzed as a water sample after a documented discussion with the laboratory.

- Remove the solid phase and filter from the filtration apparatus.
- Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value. Record the final weight.
- Calculate the percent solids as follows:

$$\frac{\text{(weight of dry waste \& filters) - (tared weight of filters)}}{\text{initial weight of waste}} \times 100 = \text{\% dry solids}$$

- If the dry solid phase comprises <0.5% of the waste, it is discarded and the liquid phase is defined as the TCLP extract. Proceed to Sec. 7.6.1.14.
- If the dry solid phase is ≥0.5% of the waste, return to Sec. 7.6.1.1 and begin the procedure with a new sample of waste. Do not extract the solid that has been dried.

7.6.1.11 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material is capable of passing through a 9.5 mm sieve, proceed to sec. 7.6.1.12. If the particle size is larger than 9.5 mm, the solid material is prepared for extraction by crushing, cutting, or grinding until it is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is ≥3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

7.6.1.12 This step describes the determination of the appropriate extracting fluid to use.

- Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0 g portion to a 250 mL beaker.
- Add 96.5 mL DI water, cover with watch glass, and stir vigorously for five minutes using a magnetic stirrer. Measure and record the pH. If the pH is ≤ 5.0, extraction fluid # 1 is used. Proceed to sec. 7.6.1.13.
- If the pH is >5.0, add 3.5 mL 1.0 N hydrochloric acid, stir for 30 seconds and heat to 50+/-5°C. Continue heating at 50+/-5°C for ten minutes.
- Let the solution cool to room temperature and record the pH. If pH is ≤ 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

7.6.1.13 Transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

NOTE: If any of the solid phase remains adhered to the walls of the filter holder, or the container used to transfer the waste, its weight shall be determined, subtracted from the weight of the solid phase of the waste, as determined above, and this weight is used in calculating the amount of extraction fluid to add into the extractor bottle.

Slowly add an amount of the appropriate extraction fluid into the extractor bottle equal to 20 times the weight of the solid phase that has been placed into the extractor bottle. Close the extractor bottle tightly, and place in the rotary extractor and rotate for 18 ± 2 hours. The ambient room temperature shall be maintained at 23 ± 2°C during the extraction period. Note: If pressure builds up in the extractor bottle, especially initially, it is permissible to vent the bottle.

7.6.1.14 Following the 18 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter as outlined in Sec. 7.6.1.7.

7.6.1.15 The TCLP extract is now prepared as follows:

- If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.1.14 is defined as the TCLP extract. Proceed to Sec. 7.6.1.16.

- If compatible (e.g., will not form a precipitate or has multiple phases), the filtered liquid is combined with the initial liquid phase of the waste. This combined liquid is defined as the TCLP extract.

- If the initial liquid phase of the waste, as obtained from Sec. 7.6.1.9 is not compatible with the filtered liquid resulting from Sec. 7.6.1.14, the liquids are not combined. The liquids are collectively defined as the TCLP extract and are analyzed separately. The results may be combined mathematically.

7.6.1.16 Measure and record the pH of the extracts and initiate an Internal Chain of Custody (ICOC) for those extracts which require it. TCLP extracts, accompanied by the extracted blank, are ready for transfer to metals digestion and/or organic extractions for further preparation according to the procedures for the particular analysis (organics or metals). *Note: For BP samples that require LaMP protocols, each analytical or prep group must also be supplied with a sufficient volume of extraction fluid to prepare an LCS.* If metals analysis is required, immediately remove and acidify (nitric acid to pH <2) an appropriate portion and reserve for analysis. Refrigerate the remaining aliquots at 4 ± 2°C.

7.6.2 Procedure for Volatiles by ZHE

The ZHE device has approximately a 500 mL internal capacity. Although a minimum sample size of 100 grams is required in Section 7.6.1, the ZHE can only accommodate a maximum 100% solids sample of 25 grams. This is due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Sec. 7.6.2.4 provides the means by which to determine the approximate sample size for the ZHE device. Although the following procedure allows for particle size reduction during the procedure, this could result in the loss of volatile compounds. If possible, any particle size reduction (see Sec. 7.6.2.5) should be conducted on the sample as it is being taken. Particle size reduction should only be conducted during the procedure if there is no other choice.

In carrying out the following steps, do not allow the waste to be exposed to the atmosphere for any more time than is absolutely necessary.

7.6.2.1 Pre-weigh the (evacuated) container which will receive the filtrate, and set it aside.

7.6.2.2 Place the ZHE piston within the body of the ZHE (it may be helpful to first moisten the piston o-rings slightly with extraction fluid). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set it aside. Set liquid inlet/outlet flange (top flange) aside.

7.6.2.3 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative 25 g sample of the waste, record the weight, and proceed to Sec. 7.6.2.5.

7.6.2.4 This sec. provides the means by which to determine the approximate sample size for the ZHE device. If the waste is liquid or multi-phasic, follow the procedure outlined in Steps 7.6.1.2 to 7.6.1.9 (using the Section 7.6.1 filtration apparatus), and obtain the percent solids by dividing the weight of the solid phase of the waste by the original sample size used. If it appears that the solid may comprise <0.5% of the waste, see below.

- Determine the percent solids by using the procedure outlined in Sec. 7.6.1.10. If the waste contains <0.5% solids, proceed to Sec. 7.6.2.7 and follow until the liquid phase of the waste is filtered using the ZHE device (Sec. 7.6.2.8). This liquid filtrate is defined as the TCLP extract and is analyzed directly.

- If the sample is $\geq$ 0.5% solids, the maximum amount of sample the ZHE can accommodate is determined by dividing 25 grams by the percent solids obtained from Sec. 7.6.2.4. Weigh out a new representative sample of the determined size by the following calculation:

$$\text{weight of waste to change ZHE} = \frac{25}{\text{percent solids}} \times 100$$

7.6.2.5 After a representative sample of the waste has been weighed out and recorded, the sample is now evaluated for the particle size (see beginning of Procedure 7.6.2). If the solid material within the waste will obviously pass through a 9.5 mm sieve, proceed immediately to Sec. 7.6.2.6. If the particle size is larger than that described above, the solid material which does not meet the above criteria is separated from the liquid phase by sieving, and the solid is prepared for extraction by crushing, cutting, or grinding if possible until the particle size is < 9.5 mm. Note: It is not necessary to cut up filamentous material such as gloves or waste filters for all the pieces to fit through the sieve if the surface area per gram is $\geq$3.1 cm^2. It is not necessary or recommended to attempt to quantify this, but the analyst should use discretion.

NOTE: Wastes and appropriate equipment should be refrigerated, if possible, to 4$\pm$2°C prior to particle size reduction. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the furthest extent possible.

When particle size has been appropriately altered, the solid is re-combined with the rest of the waste.

7.6.2.6 Waste slurries should not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly shall not be centrifuged prior to filtration. Again, this is to minimize the loss of volatile compounds to the atmosphere.

7.6.2.7 Transfer the entire sample (liquid and solid phases) quickly to the ZHE. If there is no solid/liquid separation, proceed to sec. 7.6.2.11.
Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Sec. 7.6.2.4, to determine the weight of the waste sample which will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange), and with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (more if necessary) to slowly force all headspace out of the ZHE device.

At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

7.6.2.8 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open valve. Begin applying gentle pressure of 1 - 10 psi to force the liquid phase into the filtrate collection container. If no additional liquid has passed through the filter in any two-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi.

After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any two-minute interval, proceed to the next 10 psi increment. When liquid flow has ceased, such that continued pressure filtration at 50 psi does not result in any additional filtrate within any two-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.6.2.9 The material in the ZHE is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid - but even after applying pressure filtration this material will not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% solids (see Sec. 7.6.2.4) this filtrate is defined as the TCLP extract, and is analyzed directly - proceed to Sec. 7.6.2.13.

7.6.2.10 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Sec. 7.6.2.1) from the total weight of the filtrate-filled container. The liquid phase may now be either analyzed or stored at 4 $\pm$ 2°C until time of analysis. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample (see Sec. 7.6.2.4). Record the final weight of the liquid and solid phases.

7.6.2.11 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel.

Extraction fluid #1 is used in all cases.

- With the ZHE in the vertical position, attach a connector for the extraction fluid syringe to the liquid inlet/outlet valve. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid into the ZHE. Apply pressure to the plunger to add extraction fluid into the ZHE until the amount of fluid introduced into the device equals 20 times the weight of the solid phase of the waste that is in the ZHE.

- After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the syringe & connector. Check the ZHE to make sure that all valves are in their closed positions. Pick up the ZHE and physically rotate the device in an end-over-end fashion two or three times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top.

  Put 5-10 psi behind the piston and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This is a check to show that the piston moves under 15 psi and that the o-rings are ok. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 10 ± 5 psi and check all ZHE fittings to ensure that they are closed. Document the pressure in the TCLP logbook.
- Place the ZHE in the rotary extractor apparatus and rotate the ZHE for 18 ± 2 hours. The temperature of the room shall be maintained at 23 ± 2°C during agitation.

7.6.2.12 Following the 18 hour extraction, check the pressure behind the ZHE piston by looking at the gas pressure gauge. If the pressure has not been maintained (i.e., no gas release is observed or the pressure is not within 5 psi of the initial pressure) the device is leaking. Replace ZHE o-rings or other fittings, as necessary, and re-do the extraction with a new sample of waste. The original extract can not be used. If the pressure within the device has been maintained and the final pressure is within 5 psi of the initial pressure, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container holding the initial liquid phase of the waste, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container must be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Sec. 7.6.2.8.

7.6.2.13 If the waste contained no initial liquid phase, the filtered liquid material obtained from Sec. 7.6.2.12 is defined as the TCLP extract. If the waste contained an initial liquid phase the filtered liquid material obtained from Sec. 7.6.2.12 and the initial liquid phase (Sec. 7.6.2.8) are collectively defined as the TCLP extract.

7.6.2.14 Extracts are then transferred to GC/MS Volatiles and stored in the GC/MS Volatiles cooler until analysis. Internal Chain of Custody (ICOC) procedures will be initiated for those extracts which require it.

7.7 Documentation

7.7.1 Analysis Logbook

Important extraction information including dates, temperatures, filter lot numbers, container identification numbers, etc., are recorded in the TCLP extraction logbook and completed for each day's analysis (see Attachment 2).

8.0 QUALITY CONTROL

NOTE: All quality control measures described in the appropriate analytical methods shall be followed.

8.1 QC Summary

8.1.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. The extraction fluid is to be made up daily and the pH determined and recorded within the acceptable limits.

8.1.2 A blank extraction fluid must be prepared for each type of fluid used per batch. If both extraction fluids are used, two blanks must be analyzed. The blank for the volatile analysis is the ZHE vessel filled with the extraction fluid and run through the procedure.

8.1.3 A matrix spike shall be performed for each waste type (e.g. wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. At a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.1.4 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.1.5 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than 5x the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.1.6 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2 Corrective Action

Since this is a preparation step, problems will not be known until the filtrates are analyzed. Corrective action for poor blank results will require all samples in the set to be re-prepared. Refer to the analytical SOPs for corrective actions.

9.0 DATA ANALYSIS AND CALCULATIONS

Since this is a preparatory procedure, refer to the analytical SOPs for matrix and method QC calculations.

9.1 Multiphasic Wastes with Non-compatible Liquid Phases

Determine the volume of the individual phases, analyze as appropriate, and combine the results mathematically by using a volume weighted average:

$$\text{Final Analyte Conc.} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = Volume in first phase (L)
V_2 = Volume in second phase (L)
C_1 = Conc. in first phase (mg/L)
C_2 = Conc. in second phase (mg/L)

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Streams Produced by the Method

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

Waste from this procedure will enter the "Waste Water" waste stream.

The following waste streams are produced when this method is carried out.

- Aqueous waste from the extraction step will be turned into the waste technician after the analysis has been completed on digestate. The concentration of, if present, heavy metals will dictate the disposal procedure.
- Aqueous waste that has heavy metal levels below regulatory levels will be turned into the waste technician for disposal in the "Waste Water" waste stream.
- Aqueous waste that has heavy metal levels above regulatory limits should be marked appropriately and turned into the waste technician for disposal into the "Heavy Metal Corrosive Waste Water" waste stream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to section 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Figure 1: TCLP Flowchart
Table 1: TCLP Constituents and Regulatory Levels
Attachment 1: TCLP Metals Spiking
Attachment 2: TCLP Extraction Log
Attachment 3: pH Calibration Work Instruction

14.0 REVISION HISTORY

- Revision 18, was updated on 02/06/13
- Annual Review
- Update Attachment 3

Figure 1.

TCLP Flowchart

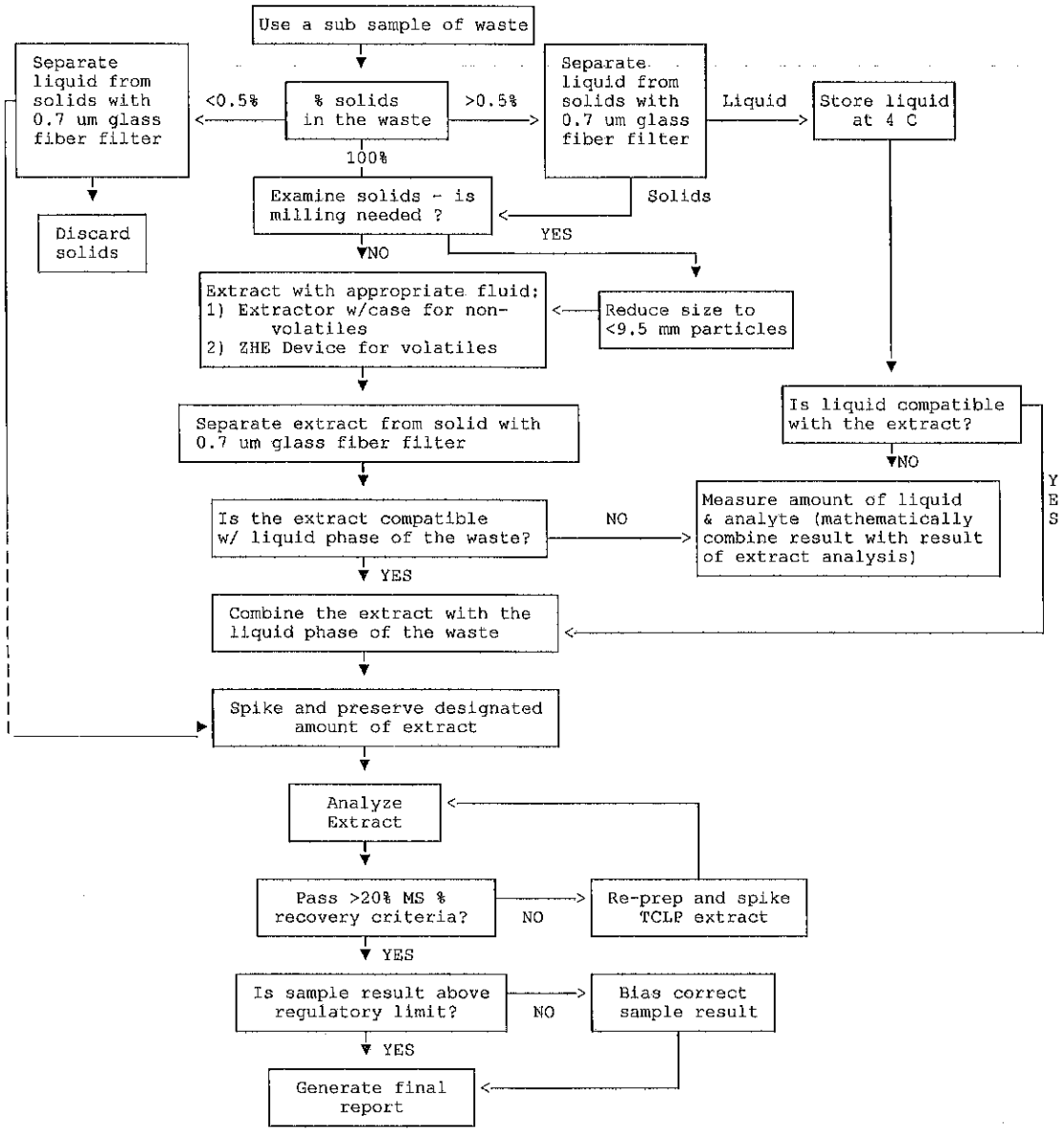

Table 1.

TCLP Constituents and Regulatory Levels

EPA HW Number	Constituent	CAS No.	Regulatory Level (ug/L)
D004	Arsenic	7740-38-2	5,000
D005	Barium	7440-39-3	100,000
D018	Benzene	71-43-2	500
D006	Cadmium	7440-43-9	1,000
D019	Carbon Tetrachloride	56-23-5	500
D020	Chlordane	57-74-9	30
D021	Chlorobenzene	108-90-7	100,000
D022	Chloroform	67-66-3	6,000
D007	Chromium	7440-47-3	5,000
D023	o-Cresol	95-48-7	*[1]200,000
D024	m-Cresol	108-39-4	*[1]200,000
D025	p-Cresol	108-44-5	*[1]200,000
D026	Cresol		*[1]200,000
D016	2,4-D	94-75-7	10,000
D027	1,4-Dichlorobenzene	106-46-7	7,500
D028	1,2-Dichloroethane	107-06-2	500
D029	1,1-Dichloroethylene	75-35-4	700
D030	2,4-Dinitrotoluene	121-14-2	130
D012	Endrin	72-20-8	20
D013	Heptachlor (& its epoxides)	76-44-8	8
D032	Hexachlorobenzene	118-74-1	130
D033	Hexachloro-1,3-butadiene	87-68-3	500
D034	Hexachloroethane	67-72-1	3,000
D008	Lead	7439-92-1	5,000
D013	Lindane	58-89-9	400
D004	Mercury	7439-97-6	200
D014	Methoxychlor	72-43-5	10,000
D035	Methyl Ethyl Ketone (2-Butanone)	78-93-3	200,000
D036	Nitrobenzene	98-95-3	2,000
D037	Pentachlorophenol	87-86-5	100,000
D038	Pyridine	110-86-1	5,000
D010	Selenium	7782-49-2	1,000
D011	Silver	7740-22-4	5,000
D039	Tetrachloroethylene	127-18-4	700
D015	Toxaphene	9001-35-2	500
D040	Trichloroethylene	79-01-6	500
D041	2,4,5-Trichlorophenol	95-95-4	400,000
D042	2,4,6-Trichlorophenol	88-06-2	2,000
D017	2,4,5-TP (Silvex)	93-72-1	1,000
D043	Vinyl Chloride	75-01-4	200

[1] If o-, m-, p-cresol concentration cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200, 000 ug/L.

Attachment 1.

TCLP Metals Spiking

The purpose of the matrix spike is to monitor the performance of the analytical methods used and to determine whether matrix interferences exist.

Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to the TCLP extraction of the sample.

In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of the TCLP extract as that which was analyzed for the unspiked sample.

The following steps detail the TCLP metals spiking procedure:

- Measure out 100 mLs of TCLP extract and transfer it into a small container.
- Using an eppendorf pipet, dispense 1 mL of each standard, STL-TCLP-1A and TCLP-2 (a.k.a., CGBA 10-A), into the TCLP extract.
- Preserve the TCLP spiked extract with 2 mLs of concentrated nitric acid.
- Store at 4 $\pm$ 2°C.

NOTE:

TCLP Stock Spike Solution Concentration:
Ba = 1000 ppm; As, Cr, Pb = 500 ppm; Cd, Se, Ag = 100 ppm.

Element Concentrations in Spiked Samples:
Ba = 100 ppm; As, Cr, Pb = 5 ppm; Cd, Se, Ag = 1 ppm

Attachment 2.

Example: TCLP Extraction Logbook

TestAmerica Chicago
TCLP Extraction Logbook

Page Number:_____

Rotator RPM : 30 ± 2 RPMs

Group Number:_____

LIMS Batch No.:_____

Sample Size Specifications:____<9.5 mm__

Sample Type (Circle): TCLV SPLP TCLP ASTM

Extraction Start Date / Time / Temp: _____/_____/_____ °C

Extraction End Date / Time / Temp: _____/_____/_____ °C

Min.Temp: Uncorrected/Corrected____/____ °C Max.Temp: Uncorrected/Corrected____/____ °C

Control Limits: 10 ± 5 PSI; 23 ± 2 °C

Filtration Start Time: _____

Filtration End Time: _____

Filter Paper Lot #: _____

Thermometer ID: _____

Sample Number					
Sample Description					
Sample Weight (g)					
Liquid-Solid Separation (Yes/No)					
Volume of Mother Liquid (mLs)					
Solid Extraction Material (g)					
Extraction Fluid Selection pH of Initial Solution: If <5.0, use Extraction Fluid #1					
pH of Acid/Heat Treated Solution: If <5.0, use Extraction Fluid #1 If >5.0, use Extraction Fluid #2					
Extraction Fluid Type (1 or 2)					
Extraction Vessel Type / Pressure Check					
Extraction Fluid Volume (mLs)					
Extract Filtered (Yes or No)					
Mother Liquid Added (mLs)					
Combined Filtrate Volume (mLs)					
Final pH Reading					
Spike Source ID # / Volume Added (mLs)					
Filtrate Preserved					
ZHE: Initial psi / Final psi					

Comments:_____

Extraction Vessel Codes: T = Teflon Organics/Metals; ZHE = Zero Headspace VOA's; HDPE = High Density Polyethylene Metals

Analyst: _____ Date:_____

Reviewer:_____ Date:_____

Attachment 3.

Example: pH Calibration Work Instruction

Calibration Instructions

Thermo Scientific: Orion Star A211 Benchtop pH Meter

C-2621

The meter **must be re-calibrated daily prior to use,** documenting the calibration in the TCLP pH logbook.

1. Bring buffers pH 2.0. 4.0, 7.0 and 10.0 to room temperature and pour fresh daily.

2. In measurement mode, press **f1 (call)**

3. Rinse the pH electrode and any other electrodes in use with distilled water, blot dry with a lint-free tissue and immerse in the pH buffer to a depth of ~1.5" in the pH 4.0 buffer.

4. When the electrode and buffer are ready, press **f3 (start)**.

5. Wait for the pH value on the meter to stabilize and stop flashing and perform one of the following actions:

a. Press **f2 (accept)** to accept the displayed pH value.

b. Press **f3 (edit)** to access the numeric entry screen and edit the pH buffer value.

i. Press **setup ∧** **mode >** **log/print >** **or** **hold <**

To highlight a number, decimal point or negative sign; **press f3 (enter)** to select the highlighted item and repeat until the buffer value at the measured temperature is shown above the numeric entry screen.

ii. Press **f2 (done)** to exit the numeric entry screen

iii. Press **f3 (accept)** to accept the entered pH value.

6. Press **f2 (next)** to proceed to the next buffer 7.0 and then 10.0 and repeat steps 3 through 5 for each then press **f3 (cal done)** to save and end calibration

7. The meter will display the calibration summary including the average slope. Press **f1 (meas)** to export the data to the calibration log. The meter will automatically proceed to the measurement mode.

8. Verify calibration by reading back the 2.0, 4.0, 7.0 and 10.0 buffers, and bracket every 10 sample readings with an alternate-source pH 7.0 QC buffer. The calibration verification is documented in the TCLP pH logbook.

For additional information, more detailed instructions, and a trouble-shooting guide, see the Instruction Manual C-2621 stored near the pH meter.

COMPANY CONFIDENTIAL AND PROPRIETARY

TITLE: SAMPLE PREPARATION
Metals Digestion by SW-846 3000 Series

Approvals (Signature/Date):

Debbie Johnson 12/14/12 Debbie Johnson Date Supervisor, Metals	Mark A Storm 12/17/12 Mark Storm Date Env. Health & Safety Coordinator
Terese A. Preston 12/17/12 Terese A. Preston Date Quality Assurance Manager	Michael J Healy 12/14/12 Michael J. Healy Date Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the guidelines for the preparation of wastewaters, extracts, wastes and soil samples for metals analysis by Trace Inductively Couples Argon Plasma (ICP) and Inductively Coupled Argon Plasma / Mass Spectrometry (ICP-MS). This SOP was written using the following methods of SW-846, Third Edition:

Method	Description
3005A	Surface and ground waters for analysis by Trace ICP, ICP-MS
3010A	Waters and extracts for analysis by Trace ICP
3030C	See note below.
3050B	Soil and waste samples for analysis by Trace ICP, ICP-MS
E 821/R-91-100	Acid Volatile Sulfide and Simultaneously Extracted Metals

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

Note: The state of North Carolina requires the use of Standard Methods 3030C for the preparation of wastewater samples.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable. Refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable. Refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

Water and soil samples are digested with nitric acid, hydrochloric acid and/or hydrogen peroxide to produce digestates that are in the correct acid media for analysis by the Trace ICP or ICP-MS.

2.0 INTERFERENCES

Matrix interferences are usually not present for the digestion process. Analytical matrix interferences may be apparent during the instrumental analysis of the digestates. The types of interferences for the instruments are discussed in the appropriate SOPs.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- Acid vapor can be dangerous. Work in a well ventilated area (i.e., fume hood).
- Hydrogen peroxide (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE:** This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. 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. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

4.0 EQUIPMENT AND SUPPLIES

- Top loading balance
- Hot plate (w/ thermometer)
- Hot Block w/ digestion vessels (w/ thermometer)
- 250 mL beakers
- 100 mL graduated cylinders
- Whatman No. 541 filter paper
- Funnels
- Fume hood(s)
- Eppendorf Pipettes
- Watch glasses (ribbed & non-ribbed)
- Filters and plunger apparatus[1]
- 100 & 50 mL digestate vessels[1] (which are checked to ensure volume markings at 25 mL, 50 mL, and 100 mL are within 2.5% Tolerance).
- 100 mL Snap-Cap containers for digestates (which are checked to ensure volume markings are within 2.5% Tolerance).

[1]Each time the metals digestions staff opens a new case/box of filters or digestion tubes, the certificate must be removed and the lot number compared with previous lot numbers. If the case/box has a new lot number, the filters or digestion tubes must be verified as free of metals contamination to the lowest reporting level by in-house testing. This will be accomplished as follows:

1. Prep analyst identifies new lot and documents the prep batch number for the first ICP and ICPMS MB created on the vendor supplied quality certificate.
2. The lot number will be documented in the comments section of the LIMs prep batch.
3. The certificate will be forwarded to the instrument analyst attached to the metals digestion sheet.
4. After the analysis of the MB, the raw data will be reviewed to determine if the acceptance criteria of results falling below ½ the current RL are met.
5. If the acceptance criteria are met, the analyst or supervisor will initial and date the certificate to indicate approval and document the analysis run and batch where the results may be found.
6. If the criteria are not met, the analyst must notify their supervisor immediately who will then initiate corrective action measures.
7. The certificate will be maintained on file in the Metals Department.

5.0 REAGENTS AND STANDARDS

5.1 Reagents

- Concentrated Nitric Acid (Instra Pure)
- Concentrated Hydrochloric Acid (Instra Pure)
- 30% Hydrogen Peroxide Solution
- 30% Ultrex II Ultra Pure Hydrogen Peroxide Solution

Purchased from a chemical vendor.

- Life of Reagent: Specified by the Manufacturer, usually 1 year.
- Storage Requirements: Acid Cabinet

5.2 Standards

5.2.1 ICP and ICP-MS Intermediate Standards

These standards are prepared from multi-element solutions purchased from vendors. Single element spikes may be used if needed. These solutions expire 1-year from the date of receipt.

Standard	Preparation
ICP Spike Solution ICP-MS Spike Solution	• Add ~400 mLs of Milli-Q water to a 1-L Class A volumetric flask. • Add 100 mLs each of HP1381-A-500, HP1381-B-500 and HP1381-C-500 (ICP); HP2930-A-500, HP2930-B-500, HP2930-C-500 (ICP-MS) • Add 9 mLs of 1,000 ppm Se; • Add 8 mLs of 1,000 ppm Pb; • Add 6 mLs of 1,000 ppm As; • Add 5 mLs of 1,000 ppm Tl; and • Add 40 mLs of InstraPure nitric acid. • Swirl to mix; Dilute to volume with Milli-Q water. Life of Standard: Expiration date of the earliest expiring standard. Storage Requirements: None.

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to SOP No. UP-SP-1311.

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

QC Indicator	Preparation	Frequency
Method Blank (MB)	For soil sample batches, use 5 - 10 mLs of Milli-Q water. [1]	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water.	1 per 20 or fewer samples.
Matrix Duplicate (DU) [2]	Aliquot of the same field sample that is digested independently.	1 per 20 or fewer samples.
Laboratory Control Sample (LCS) [3]	For soil sample batches, use 5 - 10 mLs of Milli-Q water and spike as listed below. [1 4]	1 per 20 or fewer samples.
	For water sample batches, use 50 mLs of Milli-Q water and spike as listed below. [4]	1 per 20 or fewer samples.
Matrix Spike (MS); MS Duplicate (MSD) [2]	Aliquot of the same field sample that is spiked as listed below [3] and digested independently.	1 per 20 or fewer samples.

[1] For those programs, clients, and projects mandating the use of a 'soil' matrix for the preparation of Soil MB's and LCS's (including all DoD, AFCEE and NFESC projects), Teflon Strands will be incorporated.
[2] The sample selection for MS/MSD/DU is rotated among client samples so that various matrix problems may be noted and/or addressed.
[3] LCS Duplicate (LCSD) is performed when requested by the client, contract or QAP.
[4] The LCS and MS/MSD are spiked with a known amount of analyte and processed through the digestion procedure. The spiking procedure is as follows:

Instrument	Waters Spike Volume	Soils Spike Volume
Trace ICP / ICP-MS	0.5 mL of ICP/ICP-MS Intermediate Spiking Solution.	1 mL of ICP/ICP-MS Intermediate Spiking Solution.

Refer to Appendix A for the individual element concentrations within the spiking solutions. Matrix spikes for TCLP extracts are added after filtration of the TCLP extract and before preservation. Refer to UP-SP-1311.

7.2 Sample Preservation and Storage

Matrix	Holding Time	Preservation
Waters	180 days	HNO_3, pH <2; Cool 4 ± 2°C
Soils	180 days	Cool 4 ± 2°C

Note: If the metals water samples are not preserved, they will be preserved in login and a sticker will be placed over the lid of the sample bottle. This pre-printed sticker will document "Sample preserved in login, do not analyze for 24 hours".

7.2.1 Sample Handling Procedures (Other than Soils / Waters)

Matrix	Description
Wipes	The entire wipe is digested with results reported as ug/wipe.
Paint Chips	Care is taken to remove the paint from the substrate. The chips are then cut and ground to provide a uniform matrix from which to take a sample aliquot. Sample size is approximately 1.0 grams.
Solids *	Dried and ground with a mechanical crusher.

*Bricks, wood, etc.

7.3 Sample Preparation

- Since the pH is checked by the sample custodian at sample receipt, the digestion analysis will check the pH at random and/or if the analyst has a reason to suspect that the sample may not be preserved.
- The start and end temperature of the hot plate or hot block digestion is documented in TALS.

NOTE: The LCS and MB must be filtered when analyzed with dissolved metals that are filtered in the laboratory (unpreserved samples).

7.4 Calibration / Standardization

Not Applicable.

7.5 Preventive Maintenance

- To minimize contamination during sample preparation, the fume hoods and counter areas must be kept clean and free of dust.
- The digestion hoods are cleaned on a regular basis (a minimum of once a month) and documented within the hood maintenance log.

7.6 Sample Digestion

7.6.1 Method 3005A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of InstraPure nitric acid and 2.5 mLs of InstraPure hydrochloric acid.
- Cover the vessel with a ribbed watch glass and heat on a preheated hot block at 90-95°C until the volume has been reduced to 10-15 mLs. Do not allow the samples to boil.
- Remove the vessels from the hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plates, all volumes remain the same in the 250 mL beaker. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.1.1 Method 3005A-Modified ICP-MS

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1 mL of InstraPure nitric acid and ***0.5 mLs**** of InstraPure hydrochloric acid.
- Cover the vessel with a ribbed watch glass and heat on a preheated hot block at 90-95°C until the volume has been reduced to 10-15 mLs.
- Remove the vessels from the hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using the plunger apparatus.
- The sample is now ready for analysis.

***NOTE:** Method 3005A was modified for ICP-MS to matrix match the preparation with the analytical standards. In this regard, a volume of 0.5 mL of InstraPure hydrochloric acid is added to the digestion vessel in place of 2.5 mLs. Hydrochloric acid is a known interferent on the ICP-MS.

7.6.2 Method 3010A

- Transfer 50 mLs of the well-mixed (homogenized) sample into a 50 mL digestion vessel.
- Add 1.5 mLs of InstraPure nitric acid.
- Cover the vessel with a ribbed watch glass and place on a preheated hot block set at 90-95°C.
- Evaporate the sample down to a low volume (approximately 20 mLs) – just enough to cover the bottom of the vessel. **The sample should not boil or evaporate completely on any portion of the bottom of the vessel. If this should happen, a re-digestion must be done.**
- Remove the vessel from the hot block and allow to cool.
- Add another 1.5 mLs portion of InstraPure nitric acid.

- Cover the vessel with a non-ribbed watch glass and return to the hot block to allow a gentle reflux to occur.
- Continue to add InstraPure nitric acid as necessary, until the digestion is complete (no change in appearance with continued refluxing).
- Uncover and evaporate to a low volume, not allowing any part of the vessel to go dry.
- Remove the vessels from the hot block and allow to cool.
- Add 2.5 mLs of InstraPure hydrochloric acid.
- Warm the vessel for another 15 minutes to dissolve any precipitate.
- Remove from hot block and allow to cool.
- Fill to a 50 mL final volume in the digestion vessel with Milli-Q water and filter using plunger apparatus.
- The sample is now ready for analysis.

NOTE: When using the Hot Plate, the volume remains the same in a 250 mL beaker. Transfer the digestate to a digestion vessel, washing down the sides of the beaker with Milli-Q water as needed. Dilute the sample to a 50 mL final volume using Milli-Q water and filter using the plunger apparatus.

7.6.3 Method 3030C

- Transfer 50 mLs of well mixed (homogenized) sample into a digestion vessel.
- Add 1.25 mLs of InstraPure hydrochloric acid.
- Heat 15 minutes on hot block
- Cool sample
- Filter through a 0.45 um membrane filter.
- Bring sample up to volume of 50 mL and mix.
- Sample is now ready for analysis.

7.6.4 Method 3050B

- Weigh out a portion of homogenized sample, generally 1.0 – 1.2 grams, into a 100 mL vial. The exact weight is recorded in the TALS digestion spreadsheet. More sample weight may be used if the liquid content is high, as long as the digestion is complete.

NOTE: When using the hot plate/beaker combination, soils are generally weighed to 1.00-2.00 grams. All other volumes are the same as for digestions.

- Add 5 mLs of InstraPure nitric acid and 5 mLs of Milli-Q water.
- Cover the vial with a non-ribbed watch glass and place on a preheated hotplate set at 90-95°C for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Add 5 mLs of InstraPure nitric acid and reflux for 30 minutes with a non-ribbed watch glass.
- If brown fumes are generated, repeat this last step until no brown fumes are generated indicating complete reaction with the nitric acid.
- Allow the solution to evaporate to a low volume – just enough to cover the bottom of the vial. **Do not allow the sample to boil.**
- Remove the vial from the hot block and allow to cool.
- Add 2 mLs of Milli-Q water and 3 mLs of 30% hydrogen peroxide. If for soil samples, the element Tin is included in the analytes to be reported, the 30% Ultrex II Ultra pure hydrogen peroxide must be used.
- Cover the vial with a non-ribbed watch glass and heat until the reaction is complete.

- Remove the vial from the hot block and allow to cool.
- Continue to add 30% hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance in unchanged. **Do not add more than a total of 10 mLs of hydrogen peroxide.**
- Cover the sample with a ribbed watch glass and heat until the volume has been reduced to ~5mLs or heat at 90-95°C for 2-hours without boiling.
- Maintain a covering of solution on the bottom of the vial (or beaker) at all times.

If the sample is being analyzed by ICP:

- Allow the sample to cool.
- Add 10 mLs of InstraPure hydrochloric acid.
- Place the vial on the hot block and heat for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Wash down the sides of the vial with Milli-Q water and filter into a 100 mL snap-cap container through Whatman 541 filter paper.
- Dilute the sample to the 100 mL mark in a snap-cap container.
- The sample is now ready for analysis.

If the sample is being analyzed by the ICP-MS:

- Allow the sample to cool.
- Add ***1.0 mL**** of InstraPure hydrochloric acid.
- Place the vial on the hot block and heat for 15 minutes without boiling.
- Remove the vial from the hot block and allow to cool.
- Wash down the sides of the vial with Milli-Q water and filter into a 100 mL snap-cap container through Whatman 541 filter paper.
- Dilute the sample to the 100 mL mark in a snap-cap container.
- The sample is now ready for analysis.

***NOTE:** Method 3050B was modified for ICP-MS to matrix match the preparation with the analytical standards. In this regard, a volume of 1.0 mL of InstraPure hydrochloric acid is added to the digestion vessel in place of 10 mL. Hydrochloric acid is a known interferent on the ICP-MS.

7.6.5 AVS-SEM

The SEM extracts are prepared and spiked in the wet chemistry section using 2.5 mL of the Trace (3010) spiking solution, and when requested, 2.5 mL of the 100 ug/L mercury stock, both provided by the metals digestion section. Upon receipt from Wet Chemistry, the extracts still include the solid fraction, and will need to be filtered and brought to 250 mL. The extracts are then ready for the instruments, as no digestion is required.

7.7 Documentation

7.7.1 TALS Digestion Spreadsheets

Sample digestion and standard traceability are documented within the TALS ADII spreadsheets. The spreadsheets must be completed for each day's work in real time as the samples are measured and spiked prior to putting them on the hot block. Sample IDs must be scanned into the batches to reduce transcription errors. Any notes relevant to the digestion batch must be made directly in ADII. The following information must be recorded in the Batch Notes when applicable: Filter Lot #, HCl Lot #, HNO_3 Lot #, H_2O_2 Lot #, Hot Block ID # and Temperature, Thermometer ID #, Start Time, and Digestion Tube Lot #. Refer to Appendix B for examples of digestion spreadsheets.

7.7.2 Traceability of Standards

Custom made and single element stock standard solutions which are traceable to NIST or EPA are purchased. Upon receipt, each standard is entered into the TALs LIMS database and is issued unique source ID #. The manufacturer, lot #, date received, expiration date, and the initials of the analyst are also entered.

8.0 QUALITY CONTROL

8.1 QC Summary

QC Standard	Indicator
Method Blank (MB)	Examined to determine if there was any contamination introduced during the digestion process.
Laboratory Control Sample (LCS)	Used to determine the completeness of the digestion process. The accuracy is measured by the percent recovery (%R) of each standard.
Matrix Duplicate (DU)	Demonstrate analytical precision and is reported as Relative Percent Difference (RPD).
Matrix Spike (MS) / MS Duplicate (MSD)	Used to demonstrate analytical accuracy and is reported as % recovery.

8.2 Corrective Action

A Non-Conformance Memo (NCM) will be initiated by the analyst in TALS anytime there is a deviation from the routine preparation procedures, as outlined within this SOP. This includes, but is not limited to: limited sample volume, sample loss due to spills, extremely vigorous reactions, spiking issues or any other issues that may occur during the preparatory procedure. The Section Manager will review the NCM and document any corrective action needed at that time. All analytical 'out-of-control' situations are identified as indicated in the supporting analytical SOP's proper.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

- Waste from this procedure will enter the "Corrosive Wastewater" wastestream.

11.0 METHOD PERFORMANCE CRITERIA

Refer to sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0.

13.0 ATTACHMENTS

Appendix A. Metals Digestion Standard Spike Concentrations
Appendix B. Example: ICP; ICP-MS TALs LIMS Digestion Spreadsheets

14.0 REVISION HISTORY

- Revision 22 was updated on 12/14/12
- Sections 4.0 and 7.7.1 were updated to include SOP Change form text.

Appendix A.

Metals Digestion Standard Spike Concentration

ICP

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP1381-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP1381-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, Li, V, Bi, Mn, Zn	500
		B, Fe, Sr	1,000
	HP1381-C-500	Ag	50
		Sb, P	500
		Mo, Sn, Ti	1,000
		Si	5,000
Inorganic Ventures	Single Element Standard	As	1,000
		Pb	1,000
		Se	1,000
		Tl	1,000

ICP-MS

Vendor	Stock Name	Elements	Conc. (mg/L)
Environmental Express	HP2930-A-500	Al, Ba	2,000
		Ca, Mg, K, Na	10,000
	HP2930-B-500	Se	10
		Pb	20
		As	40
		Tl, Be, Cd	50
		Cr	200
		Cu	250
		Co, Ni, V, Mn, Zn	500
		B, Fe, Sr	1,000
	HP2930-C-500	Ag	50
		Sb	500
		Mo, Sn, Ti	1,000
		Si	5,000
Inorganic Ventures	Single Element Standard	As	1,000
		Pb	1,000
		Se	1,000
		Tl	1,000

TCLP (MS)

Vendor	Stock Name	Elements	Conc. (mg/L)
Inorganic Ventures	STL-TCLP-1A	Hg	25
		Cu	25
		Zn, Ni	50
		Cd, Se, Ag	100
		Cr, As, Pb	500
	Single Element Standard	Ba	10,000

AVS-SEM (LCS)
(10 g/250 mL)

Elements	Conc. (mg/kg)
As	2.5
Cd	1.25
Cr	5.0
Cu	6.25
Pb	2.5
Ni	12.5
Ag	1.25
Zn	12.5

Appendix B.

Example: TALs LIMS Digestion Spreadsheets (014-001 to 014-010)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172669 | Analyst: Laird, Rebecca | Batch Open: 12/11/2012 3:30:00PM

Method Code: 500-3005A-500 | Batch End: 12/11/2012 4:00:00PM

Preparation, Total Recoverable or Dissolved Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments
1	MB 500-172669/1	MB~500-172669/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
2	LCS 500-172669/2	LCS~500-172669/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
3	500-53056-A-12-A	500-53056-A-12-A (6020)	N/A	Filtrate	50 mL	50 mL	12/13/12	4_Days	N/A	
4	500-53135-A-1	500-53135-A-1 (6020)	N/A	Water	50 mL	50 mL	12/19/12	6_Days - R	N/A	
5	500-53135-A-1 DU	500-53135-A-1~DU (6020)	N/A	Water	50 mL	50 mL	12/19/12	6_Days - R	N/A	
6	500-53135-A-1 MS	500-53135-A-1~MS (6020)	N/A	Water	50 mL	50 mL	12/19/12	6_Days - R	N/A	
7	500-53135-A-1 MSD	500-53135-A-1~MSD (6020)	N/A	Water	50 mL	50 mL	12/19/12	6_Days - R	N/A	

(014-001)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172669 | Analyst: Laird, Rebecca | Batch Open: 12/11/2012 3:30:00PM

Method Code: 500-3005A-500 | Batch End: 12/11/2012 4:00:00PM

Batch Notes

Digestion Tube/Cup Lot #	127143
Hot Block ID number	9
Hood ID or number	
Lot # of Nitric Acid	L08022
Lot # of hydrochloric acid	0000007153
Uncorrected Temperature	90
Oven, Bath or Block Temperature 1	
ID number of the thermometer	
Uncorrected Temperature 2	90
Oven, Bath or Block Temperature 2	
Filter Paper Lot Number	
Pipette ID	1627
Person who witnessed spiking	
First Start time	1530
First End time	1600
pH Paper Lot Number	
Batch Comment	

(014-002)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172669 | Analyst: Laird, Rebecca | Batch Open: 12/11/2012 3:30:00PM

Method Code: 500-3005A-500 | Batch End: 12/11/2012 4:00:00PM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-172669/2	M12KSPKMS_00001	0.5 mL	50 mL		
500-53135-A-1 MS	M12KSPKMS_00001	0.5 mL	50 mL		
500-53135-A-1 MSD	M12KSPKMS_00001	0.5 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(014-003)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172801 | Analyst: Laird, Rebecca | Batch Open: 12/12/2012 3:00:00PM

Method Code: 500-3010A-500 | Batch End: 12/12/2012 3:30:00PM

Preparation, Total Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments
1	MB 500-172801/1	MB~500-172801/1 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
2	LCS 500-172801/2	LCS~500-172801/2 N/A	N/A		50 mL	50 mL	N/A	N/A	N/A	
3	500-53164-J-1	500-53164-J-1 (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
4	500-53164-J-2	500-53164-J-2 (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
5	500-53164-J-3	500-53164-J-3 (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
6	500-53164-J-3 DU	500-53164-J-3~DU (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
7	500-53164-J-3 MS	500-53164-J-3~MS (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
8	500-53164-J-3 MSD	500-53164-J-3~MSD (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
9	500-53164-J-4	500-53164-J-4 (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	
10	500-53164-J-5	500-53164-J-5 (6010B)	N/A	Water	50 mL	50 mL	12/31/12	12_Days - R	N/A	

(014-004)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172801 | Analyst: Laird, Rebecca | Batch Open: 12/12/2012 3:00:00PM

Method Code: 500-3010A-500 | Batch End: 12/12/2012 3:30:00PM

Batch Notes

Lot # of hydrochloric acid	0000007153
Lot # of Nitric Acid	L08022
Vendor of Reagent used	
Digestion Tube/Cup Lot #	20313760
Hot Block ID number	4
ID number of the thermometer	J59688
Uncorrected Temperature	90
Oven, Bath or Block Temperature 1	
Uncorrected Temperature 2	90
Oven, Bath or Block Temperature 2	
Filter Paper Lot Number	
Pipette ID	1627
Person who witnessed spiking	
First Start time	1500
First End time	1530
Batch Comment	

Comments

(014-005)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172801 | Analyst: Laird, Rebecca | Batch Open: 12/12/2012 3:00:00PM

Method Code: 500-3010A-500 | Batch End: 12/12/2012 3:30:00PM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-172801/2	M12JSPKIC_00002	0.5 mL	50 mL		
500-53164-J-3 MS	M12JSPKIC_00002	0.5 mL	50 mL		
500-53164-J-3 MSD	M12JSPKIC_00002	0.5 mL	50 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(014006)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172542 | Analyst: Hudson, Lawrence A | Batch Open: 12/11/2012 7:20:00AM

Method Code: 500-3050B-500 | Batch End: 12/11/2012 7:50:00AM

Preparation, Metals

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	Matrix	Initial Amount	Final Amount	Due Date	Analytical TAT	Div Rank	Comments
1	MB 500-172542/1	MB~500-172542/1 N/A	N/A		1 g	100 mL	N/A	N/A	N/A	
2	LCS 500-172542/2	LCS~500-172542/2 N/A	N/A		1 g	100 mL	N/A	N/A	N/A	
3	500-53114-A-1	500-53114-A-1 (6010B)	N/A	Solid	1.0546 g	100 mL	12/12/12	2_Days - R	N/A	
4	500-53114-A-2	500-53114-A-2 (6010B)	N/A	Solid	1.0263 g	100 mL	12/12/12	2_Days - R	N/A	
5	500-53114-A-3	500-53114-A-3 (6010B)	N/A	Solid	1.0356 g	100 mL	12/12/12	2_Days - R	N/A	
6	500-53114-A-3 DU	500-53114-A-3~DU (6010B)	N/A	Solid	1.0241 g	100 mL	12/12/12	2_Days - R	N/A	
7	500-53114-A-3 MS	500-53114-A-3~MS (6010B)	N/A	Solid	1.1139 g	100 mL	12/12/12	2_Days - R	N/A	
8	500-53114-A-3 MSD	500-53114-A-3~MSD (6010B)	N/A	Solid	1.1564 g	100 mL	12/12/12	2_Days - R	N/A	

(014007)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172542 | Analyst: Hudson, Lawrence A | Batch Open: 12/11/2012 7:20:00AM

Method Code: 500-3050B-500 | Batch End: 12/11/2012 7:50:00AM

Batch Notes

Perform Calculation (0=No, 1=Yes)	
Nominal Amount Used	
Digestion Tube/Cup Lot #	MF06LKK01
Blank Soil Lot Number	
Balance ID	C-1966
Hood ID or number	3
Hot Block ID number	8
Lot # of hydrochloric acid	0000007153
Lot # of Nitric Acid	L08022
Logbook ID for diluted Nitric	
Hydrogen peroxide lot number	L08A06
ID number of the thermometer	J59717
Temperature	
Filter Paper Lot Number	
Acid used for pH adjustment	
Pipette ID	1628
Analyst	LH
First Start time	0720
First End time	0750
Person's name who witnessed reagent drop	MP

(80470)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172542 | Analyst: Hudson, Lawrence A | Batch Open: 12/11/2012 7:20:00AM

Method Code: 500-3050B-500 | Batch End: 12/11/2012 7:50:00AM

Uncorrected Temperature	
Oven, Bath or Block Temperature 1	90
Uncorrected Temperature 2	
Oven, Bath or Block Temperature 2	90
Batch Comment	

Comments

(600-410)

Metals/Inorganics Prep Worksheet

(Used for Collecting Prep Info)

Batch Number: 500-172542 | Analyst: Hudson, Lawrence A | Batch Open: 12/11/2012 7:20:00AM

Method Code: 500-3050B-500 | Batch End: 12/11/2012 7:50:00AM

Reagent Additions Worksheet

Lab ID	Reagent Code	Amount Added	Final Amount	By	Witness
LCS 500-172542/2	M12KSPKIC_00001	1 mL	100 mL		
500-53114-A-3 MS	M12KSPKIC_00001	1 mL	100 mL		
500-53114-A-3 MSD	M12KSPKIC_00001	1 mL	100 mL		

Other Reagents:

Reagent	Amount/Units	Lot#:

(014-010)

TITLE: Sample Preparation Semivolatile and Nonvolatile Organic Compounds from a WasteWater or Leachate Matrix using Separatory Funnel Extraction

Approvals (Signature/Date):

2/21/13	2/21/13
Daniel A. Knieriemen — Date Supervisor, Organic Extractions	Mark Storm — Date Env. Health & Safety Coordinator
2/21/13	2/25/13
Terese A. Preston — Date Quality Assurance Manager	Michael J. Healy — Date Laboratory Director

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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) outlines the extraction procedure of semivolatile and nonvolatile organic compounds from wastewater or leachate matrices using SW-846, Third Edition, Methods 1311, 3500B and 3500C, and 3510C; and 40CFR, Part 136, Methods 608, 610, and 625 as references.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable - refer to the analytical SOPs.

1.1.2 Reporting Limits

Not Applicable - refer to the analytical SOPs.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

The following procedure is used for the separatory funnel extraction of semivolatile and nonvolatile organics from waste water and leachate matrices for subsequent analysis.

A measured volume of sample, usually 1-liter for waste waters, 100 mLs for leachates and with the addition of a 125 to 250 mL sample size for the Low Volume Sampling Initiative - Large Volume Injection Instruments (LVI) at the appropriate pH is serially extracted with Dichloromethane (DCM, a.k.a., Methylene chloride) using the separatory funnel extraction procedure. The extract is dried, concentrated, exchanged to another solvent if necessary, adjusted to the appropriate final volume, and stored at 4 ± 2°C prior to analysis. Optional cleanup procedures may be performed upon request. The mandatory acid cleanup for PCB samples is described within this SOP. GPC, Florisil, sulfur removal cleanups, and silica gel cleanup are described in UP-SP-003.

2.0 INTERFERENCES

- 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.
- 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 material.
- Soap residue on glassware may cause degradation of certain analytes. This problem is especially pronounced with glassware that may be difficult to rinse. These items should be hand-rinsed very carefully to avoid this problem.
- Matrix interferences may be caused by contaminants that are co-extracted from the sample. These can appear as large, distinct peaks and/or elevated baselines. Occasionally matrix interferences may prevent the proper detection of surrogate and/or analytes, resulting in the reporting of low, or possibly high, surrogate and/or spike recoveries. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the site being sampled. Various cleanup procedures may be performed to remove heavy background or interferences, including GPC cleanup, florisil cleanup, acid cleanup, silica gel cleanup, and sulfur removal procedures.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, PCBs, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, 4,4'-DDT, dibenz(a,h)anthracene and N-nitrosodimethylamine. Primary standards of these toxic compounds should be prepared in hood.
- The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.
- Nitrile or equivalent gloves are available and must be worn when handling acids, bases, or samples.
- All extractions and handling of solvents should be performed under well-ventilated conditions of a fume hood.
- When pouring large volumes of solvents (100 mLs or greater), using a squeeze bottle, using the Cycletainer toggle, or transferring solvent in any manner such that a splash can occur, safety glasses are inadequate to protect the eyes and face. A face shield must be worn. A hood sash can act as a face shield when working with solvents inside a fume hood.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause Irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

There are no materials used in this method that have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

4.0 EQUIPMENT AND SUPPLIES

4.1 Glassware

- 2 Liter separatory funnels
- 250 mL, 500 mL Erlenmeyer flasks
- Long stem glass funnels
- 100 mL, 1,000 mL graduated cylinder
- 500 mL Kuderna-Danish Flask
- 10 mL graduated receivers with attachments, regular and insulated types
- 3-Ball Snyder column
- 1.0 mL, 10.0 mL, class A, volumetric pipet
- 2.0 mL disposable pipet
- 500 uL, 1,000 uL syringe
- 5.0 mL, 10.0 mL pre-marked 16 X 125 test tubes with Teflon lined lids*
- Glass stirring rods
- Shallow Pyrex drying tray
- Teflon squeeze bottles
- Teflon centrifuge bottles, 125 mL

*Volumetrically add the appropriate volume of Hexane to a 16 X 125 screw top test tube and mark the meniscus using a fine-tipped marker. Discard the Hexane into an appropriate waste vessel and let dry. Dilute the sample to volume using this test tube.

4.2 Miscellaneous

- glasswool, soxhlet-extracted using DCM
- transfer pipets
- wide range pH paper 1 to 12
- Centrifuge
- Boiling Chips, Teflon, soxhlet-extracted using DCM
- Muffle furnace
- Desiccator
- Balance, top-loading, capable of weighing to 0.001g
- Waterbath, heated, with concentric ring covers, capable of temperature control - Must be in a hood
- Nitrogen Evaporator - N-Evap by Organomation Associates, Inc.

5.0 REAGENTS AND STANDARDS

A label on any reagent or standard bottle must contain the standard number, concentration of the reagent, name of the reagent, date prepared, expiration date and the analyst who prepared the reagent.

5.1 Reagents

5.1.1 Pesticide Grade Methylene Chloride [Dichloromethane (DCM)]
Pesticide Grade Acetone
Pesticide Grade Hexane
Pesticide Grade Acetonitrile

5.1.2 Heat Purified Granular Sodium Sulfate

Purify by heating to 400°C for 4-hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

5.1.3 Reagent Water

Defined as water in which no interferent is observed at one-half the reporting limit of any target compounds when one liter of the reagent water is extracted and prepared by using the same procedure as for a water sample.

5.1.4 Concentrated Sulfuric Acid

Purchased from a supplier

- Hazardous Properties: Sulfuric Acid (H_2SO_4) is extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year
- Storage Requirements: Store in glass container. The label on the bottle must contain concentration and name of reagent.

5.1.5 Sulfuric Acid (1:1)

Cautiously add 500 mLs concentrated sulfuric acid to 500 mLs reagent water. Because this is an exothermic reaction, prepare in Pyrex glassware and in a cold water bath.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year from preparation date
- Storage Requirements: Store in a glass bottle

5.1.6 10 N Sodium Hydroxide

Cautiously dissolve 400 grams of solid NaOH in reagent water and dilute to 1000 mLs. Always prepare in Pyrex glassware due to the amount of heat given off by the reaction.

- Hazardous Properties: Toxic & Corrosive. Harmful if swallowed, inhaled or absorbed through the skin.
- Life of Reagent: 1-year from preparation date
- Storage Requirements: Store in a glass bottle.

5.1.7 50% Hydrochloric Acid

Cautiously add 500 mLs concentrated hydrochloric acid to 500 mLs reagent water. Because this is an exothermic reaction, prepare in Pyrex glassware and in a cold water bath.

- Hazardous Properties: Extremely corrosive and toxic to tissues. Vapors are also harmful.
- Life of Reagent: 1-year from preparation date
- Storage Requirements: Store in a glass bottle

5.2 Quality Control (QC) Solutions

QC solutions must be stored in amber or foil-wrapped, glass containers that have Teflon-lined caps. The label on the container must contain the following information: Name of the solution, the standard number, concentration of the components, date prepared, preparation analyst, and expiration date. All QC solutions are stored in the standards refrigerator in the GC/MS lab, GC Extractables lab, or in the standards refrigerator located in the organic extraction lab. QC solutions are stored at 4 ± 2°C.

Listed below are the various QC solutions and concentrations appropriate for extraction of BNAs, Pesticides, Pesticides/PCBs, PCBs, PAHs, and diesel range organics (DRO) from waste water and TCLP Leachate samples.

5.2.1 Surrogate Spike Solutions

5.2.1.1 BNA Surrogate Spike Working Solution

The BNA Surrogate Spike Stock Solution is purchased directly from a chemical vendor at 10 times the concentration of the levels listed below. See Attachment 3. The BNA Surrogate Spike Working Solution is prepared by pipetting 10.0 mLs of BNA Surrogate Spike Stock Solution to a 100.0 mL flask. Class A glassware must be used. The standard is then brought up to 100.0 mLs with methanol. The BNA Surrogate Spike Solution is added to all samples, method blanks (MBs), matrix spike/matrix spike duplicates (MS/MSDs) and laboratory control samples (LCS).

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	100
Nitrobenzene-d_5	100
2-Fluorobiphenyl	100
1,2-Dichlorobenzene-d_4	100
Phenol-d_5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d_4	150

- Life of Standard: 6 months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

5.2.1.2 BNA Surrogate (SIM) Spike Solution

The BNA Surrogate (SIM) Spike Solution is prepared from the BNA Surrogate Spike Solution. The BNA Surrogate (SIM) Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	10
Nitrobenzene-d_5	10
2-Fluorobiphenyl	10
1,2-Dichlorobenzene-d_4	10
Phenol-d_5	15
2-Fluorophenol	15
2,4,6-Tribromophenol	15
2-Chlorophenol-d_4	15

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2

5.2.1.3 BNA Low Level Surrogate Spike Working Solution

The BNA Low Level Surrogate Spike Working Solution is prepared from the BNA Surrogate Spike Solution. The BNA Surrogate Working Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	50
Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
1,2-Dichlorobenzene-d_4	50
Phenol-d_5	75
2-Fluorophenol	75
2,4,6-Tribromophenol	75
2-Chlorophenol-d_4	75

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2

5.2.1.4 Pesticide/PCB Surrogate Spike Parent Solution

A certified stock solution is purchased directly from a standards vendor. The GC Extractables lab prepares a surrogate parent solution from these standards and is a solution of decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCX) in acetone at the concentrations listed below. Refer to the analytical SOP for specific details on the preparation, storage and labeling of these solutions.

Compound	Concentration (ug/mL)
DCB	4.0
TCX	4.0

- Life of Standard: 6 months from date of preparation, not to exceed expiration date of standards
- Storage Requirements: As stated in Section 5.2

5.2.1.5 Pesticide/PCB Surrogate Spike Solution

The surrogate used in the extraction of pesticides/PCBs from waste waters is a solution of DCB and TCX in acetone at the concentrations listed below. The working solution is prepared from the parent solution by performing a 1/10 dilution using Class A volumetric glassware in acetone. The Pesticide/PCB Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
DCB	0.40
TCX	0.40

- Life of Standard: 6 months from date of preparation, not to exceed expiration date of the parent standard
- Storage Requirements: As stated in Section 5.2

5.2.1.6 PAH Surrogate Spike Solution

The surrogate used in the extraction of PAHs from soil/sediments is a solution of Decafluorobiphenyl and Benzo (e) pyrene in acetone at the concentrations listed below. The HPLC group prepares the working surrogate solution. The PAH Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
Decafluorobiphenyl	100
Benzo (e) pyrene	5.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.1.7 DRO Surrogate Spike Solution

The surrogate used in the extraction of DRO from soil/sediments is a solution of 2-Fluorobiphenyl and o-Terphenyl in acetone at the concentrations listed below. The GC group prepares the working surrogate solution. The DRO Surrogate Spike Solution is added to all samples, the MB, MS/MSD and LCS.

Compound	Concentration (ug/mL)
2-Fluorobiphenyl	200
o-Terphenyl	200

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2 Laboratory Control Sample (LCS) & Matrix Spike (MS) Solutions

5.2.2.1 BNA TCL Spike Solution

The BNA TCL Spike Solution is prepared from two stock standards, 8270 LCS Mix and 3,3'-Dimethylbenzidine. See Attachments 4 and 5. 50.0 mLs of 8270 LCS Mix and 5.0 mLs of 3,3'-Dimethylbenzidine are pipetted into a 100.0 mL flask. Class A glassware must be used. The standard is then brought up to 100.0 mLs with methanol. The BNA TCL Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
Acid Compounds	100
Base/Neutral Compounds	100

- Life of Standard: 6 months from the from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2.

5.2.2.2 BNA Matrix Spike (SIM) Solution

The BNA Matrix Spike (SIM) Solution is prepared from the BNA TCL Spike Solution found in section 5.2.2.1. The BNA Matrix Spike (SIM) Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acid Compounds	10
Base/Neutral Compounds	10

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.3 BNA Low Level Matrix Spike Solution

The BNA Low Level Matrix Spike Solution is prepared from the BNA TCL Spike Solution found in section 5.2.2.1. The BNA Low Level Matrix Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acid Compounds	50
Base/Neutral Compounds	50

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.4 Pesticide (Full List) Spike Solution

The LCS/MS solution used in the extraction of pesticides or pesticides/PCB from soil/sediments is a solution of pesticide compounds in methanol at the following concentrations. The Pesticide (Full List) Spike Solution is added to MS/MSD and LCS.

Compound	Concentration (ug/mL)
All compounds	0.40

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.5 PCB Spike Solution

The LCS / MS spike solution used in the extraction of PCBs from waste waters is a solution of Arochlor 1016 and Arochlor 1260 in methanol at the following concentrations. The PCB Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
AR1016	5.0
AR1260	5.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.6 TCLP Pesticide Spike Working Solution

The LCS / MS spike solution used in the extraction of pesticides from TCLP leachates is a solution of pesticide compounds in methanol at the following concentrations. The TCLP Pesticide Spike Solution is added to the MS and LCSs that are for Pesticides only

Compound	Concentration (ug/mL)
Lindane	0.1
Heptachlor	0.1
Heptachlor Epoxide	0.1
gamma-Chlordane	0.1
Endrin	0.1
Methoxychlor	1.0

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.7 Toxaphene Spike Working Solution

The LCS / MS spike solution used in the extraction of pesticides from TCLP leachates is a solution of toxaphene in methanol at the following concentration. The Toxaphene Spike Solution is added to the LCS and MSs that are for PCBs only.

Compound	Concentration (ug/mL)
Toxaphene	10

- Life of Standard: Same as parent solution
- Storage Requirements: As stated in Section 5.2

5.2.2.8 PAH Spike Solution

The LCS/MS solution used in the extraction of PAHs from soil/sediments is a solution of analytes in acetonitrile at the following concentrations. The HPLC group prepares the spike solution. The PAH Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Acenaphthene	8
Acenaphthylene	16
Anthracene	0.8
Benzo(a)anthracene	0.8
Benzo(a)pyrene	0.8
Benzo(b)fluoranthene	1.6
Benzo(ghi)perylene	1.6
Benzo(k)fluoranthene	0.8
Chrysene	0.8
Dibenzo(a,h)anthracene	1.6
Fluoranthene	1.6
Fluorene	0.8
Indeno(1,2,3-cd)pyrene	0.8
Naphthalene	8.0
Phenanthrene	0.8
Pyrene	0.8

- Life of Standard: Same as the parent solution.
- Storage Requirements: As stated in Section 5.2.

5.2.2.9 DRO Spike Solution

The LCS/MS solution used in the extraction of DRO from soil/sediments is a solution Diesel Fuel #2 at 1000 ug/mL in methanol. The GC group prepares the spike solution. The DRO Spike Solution is added to the MS/MSD and LCS.

Compound	Concentration (ug/mL)
Diesel Fuel #2	4,000

- Life of Standard: Same as the parent solution.
- Storage Requirements: As stated in Section 5.2.

6.0 CALIBRATION

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

The following QC is performed with each organic extraction batch. An extraction batch contains samples of similar matrix that are logged in for the same test code. An extraction batch may contain no more than 20 samples.

Quality Control	Frequency
MB	1 in 20 or fewer samples
LCS [1]	1 set of 20 or fewer samples
MS/MSD [2]	At least one set in 20 or fewer samples (may be requested by client)
Surrogates	Every sample / MB / MS / MSD / LCS

[1] LCS Duplicate (LCSD) is performed when insufficient sample is available for the MS/MSD or if requested by the client.
[2] The sample selection for MS/MSD is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Collection, Preservation and Handling

Sample container, preservation technique and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract/client requests. In general, water samples are collected in 1-liter or 80-oz. narrow-mouth, amber, glass jars, with Teflon-lined screw-cap lids. If a Teflon-lined lid is not available, solvent-rinsed aluminum foil can be used as a liner.

NOTE: If the sample matrix is highly acidic or basic, corrosion of the foil may occur and contaminate the sample.

Matrix	Holding Time (VTS) [1]	Preservation
Wastewaters	7 days	Cool, 4 ± 2°C
TCLP Leachates	14 days to TCLP Extract [2] 7 days from the completion of the TCLP extraction for the organic extraction	Cool, 4 ± 2°C

[1] Verified Time of Sampling.
[2] TCLP Extraction performed by Inorganic Prep Group.

Samples, sample extracts and standards are stored separately. Samples are stored at 4 ± 2°C prior to extraction. Any samples received by the preparation group that do not comply must be noted. Deviations in results "could" be explained by the failure of sample collector/client/custodian to comply to sample collection and storage requirements.

7.3 Sample Preparation (Glassware Preparation)

7.3.1 All glassware (except Snyder columns, volumetric flasks, volumetric pipets, and syringes) must be washed thoroughly with warm soapy water, rinsed **5** times with hot tap water and rinsed **3** times with DI water. The stopcocks of the separatory funnels should be cleaned with a small brush. **It is absolutely mandatory that all glassware be scrupulously clean.** Failure to rinse well with water, may leave a soap film that is incompletely removed with solvent rinsing, and with subsequent extraction, "soap" peaks will appear in the chromatograms.

7.3.2 If glassware is very wet it may be towel dried. **All** Glassware should be rinsed with cleaning grade acetone, and the excess should be discarded into the appropriate waste container. This step is performed to ensure that the glassware is dry. **If after rinsing once, the glassware still contains any water, repeat the acetone rinse.** At this point let the glassware dry before continuing to ensure that all of the water is gone. All glassware should then be rinsed with DCM using a Teflon squeeze bottle, and the excess discarded into the appropriate waste container. Repeat this step at least 3-times and allow the glassware to dry. Failure to remove all water prior to DCM rinsing prevents the DCM solvent from coming into contact with all surfaces on the glassware. The solvent rinse is then incomplete, and may leave contaminants on the glass, which are then co-extracted with the sample.

NOTE: DCM is considered a health hazard. It is harmful if swallowed or inhaled. The vapor is irritating and thus should be used in a hood. It is readily absorbed through the skin and contact may be irritating.

NOTE: Pressure builds up quickly in a capped separatory funnel. Vent the pressure immediately and often thereafter.

7.3.3 Procedures for Return of Glassware to Dishroom

Any glassware that has organic residue present, all K-D receivers, and all separatory funnels should be rinsed with cleaning grade acetone, or whatever other solvent which removes the residue, prior to return to the dish room from the lab. Glassware in which the residue can not be removed should be segregated from other glassware being returned to the dish room. Glassware should be carefully placed in a tub and placed on the "Dirty" side of the dish racks. In order to reduce glassware breakage, glassware should not be stacked in the tubs. Severely chipped glassware should be removed from service. Some items are repairable. Check with the supervisor as to what items get repaired.

7.4 Calibration/Standardization

Not Applicable.

7.5 Preventive Maintenance

- Balance should be cleaned and checked for levelness prior to each use. Class "S" weights should be weighed and documented each day the balance is used. Consult your supervisor if the weights are out of acceptable limits. Do not use the balance until the situation is back in control.
- The K-D water bath should contain DI water only. The water level should be checked prior to each use, and should not fall below the heating coils. If the heating coils do become exposed to the air while in use, they could burn-out and would require replacement. Keep the water level approximately one inch from the top of the bath edge.
- The N-Evap water bath should also contain DI water. The water level in the bath should be sufficient to cover 3/4 of the receivers when submerged.
- The thermometer used in the extraction water baths are checked against a reference NIST thermometer on a yearly basis or sooner if necessary.
- The desiccant used in the desiccator should be removed and dried in an oven at 130°C whenever the color indicates that it is saturated with moisture. This is usually indicated by a change of color from blue to purple/pink.
- To avoid the breakage of sample extracts, the analyst needs to take care that the containers are properly balanced on the centrifuge. The container should fit snugly in the holder. Use a paper towel to cushion the centrifuge bottles if necessary.

7.6 Extraction Process

7.6.1 MB and LCS Water Preparation

Add 1.0 liter (125 or 250 mLs for LVI) of Milli-Q water to 2 separatory funnels and label as the MB, LCS and LCSD (if the batch does not contain an MS/MSD.)

7.6.2 Sample Preparation

Prior to pouring water samples, sample containers should be inverted a few times to mix the sample. However, if there is a sediment layer present, consult your supervisor or section manager before mixing. Also, if the sample appears to have more than one phase, immediately contact your supervisor, section manager, or project manager. The client will need to be notified.

a. Waste Waters (1-liter sample size, 100-250 mL for LVI)

In order to more accurately and easily resolve the initial volume of a water sample, gravimetric determination will be used. For this purpose it will be assumed that the density of the water sample is equal to 1 g/mL. To determine the initial volume, the full sample bottle is weighed and then the sample is poured into a separatory funnel. Next, the empty bottle is weighed and the value is subtracted from the full sample bottle weight. The result equals the initial volume.

Use the Mettler Toledo top loader balance. It must be cleaned and checked for levelness prior to each use. Class "S" weights must be weighed and documented each day the balance is used. Consult your supervisor if the weights are out of acceptable limits. Do not use the balance until the situation is back in control.

Create a prep batch using the 3510C_IVWT method. Scan in the samples into the batch on the Sample List tab. When the weights are ready to be entered click on the Worksheet tab. Sample weights are electronically transferred to the TALs prep batch and are recorded with one decimal place. Weigh the full sample bottle and return the result to the Gross Weight column.

Pour the sample into the separatory funnel. When all of the samples have been poured out and the bottles have been rinsed with the first aliquot of the extraction solvent, weigh the empty bottles. Return these values into the Tare Weight column. TALS will perform the subtraction and record the result in the Initial Amount column automatically.

For LVI samples add 6 g of NaCl, and then shake to dissolve the salt.

Rinse the bottle with 60 mls (30 mLs for LVI) of DCM (the first aliquot of the extraction), and add it to the extraction vessel. This ensures that the sample is qualitatively transferred.

The following procedure is one alternative to determine the initial volume. If the sample container is 1-liter, 1-quart, 125, or 250 mL in volume, mark the level on the outside of the bottle. Pour the entire contents of the bottle into the separatory funnel. Refill the bottle to the mark with water and then measure the volume in the bottle. If a bottle is full with sample, the volume still needs to be measured due to variations from bottle to bottle. Record this amount as the initial sample volume. Occasionally the analyst will encounter samples that contain sediment which has collected at the bottom of the sample bottle. The bottle still must be rinsed with DCM and the DCM/sediment transferred to the separatory funnel. If a sample contains an unreasonable amount of sediment (1/4 inch or more) contact the supervisor before continuing.

Additionally, as a second alternative, a graduated cylinder may be used to determine the initial volume. If a graduated cylinder is used, both it and the sample bottle must be rinsed with the first aliquot of DCM.

Use 1.0 liter aliquots for MS/MSD (125-250 mL for LVI) if sample volume allows. **Note: Splitting of sample volume to perform a MS/MSD is not allowed for DoD QSM, BP LaMP, and South Carolina DHEC projects.** Label as the sample, MS and MSD.

b. TCLP Leachates

For BNA TCLP samples, measure 900 mLs of Milli-Q water into each sample separatory funnel. Measure 100 mLs of each sample and add it to the labeled separatory funnel containing the water.

For Pesticide TCLP samples, measure 990 mLs of Milli-Q water into each sample separatory funnel. Measure 10 mLs of each sample and add it to the labeled separatory funnel containing the water.

Measure an additional aliquot of sample to those designated to have a MS. LCSD and MSD are not performed.

7.6.3 Check the pH by dipping a transfer pipet into the sample and touching it to wide-range pH paper. Record the pH in the extraction log.

7.6.4 Add the surrogate spike solution to every sample including the MB, MS/MSD and LCS. Refer to the flowchart in Attachment 2 for the appropriate amount to add. Note: For DoD QSM Projects the surrogate spike must be added to the sample bottle prior to transfer to the separatory funnel. If the sample bottle is completely filled, an aliquot of sample may be transferred to the separatory funnel prior to spiking. Samples which contain sediments that are required to be decanted will be spiked after transfer to the separatory funnel.

7.6.5 Add the spike solution to the LCSs and MSs. Refer to the flowchart in Attachment 2 for the appropriate amount to add. Note: For DoD QSM Projects the surrogate spike must be

added to the sample bottle prior to transfer to the separatory funnel. If the sample bottle is completely filled, an aliquot of sample may be transferred to the separatory funnel prior to spiking. Samples which contain sediments that are required to be decanted will be spiked after transfer to the separatory funnel.

NOTE: All spiking solutions MUST be at room temperature before use. This includes stock solutions when preparing working spike solutions. Actual concentrations will change at colder temperatures. Furthermore, some components may come out of solution in the freezer. Warming to room temperature reverses the process.

7.6.6 Adjust the pH as required. Refer to Flowchart in Attachment 2 for the appropriate pH for each parameter. Use sulfuric acid (1:1) to acidify and NaOH (10N) to make basic. Record pH adjustments in the log book. **Note: Use the 50% hydrochloric acid to acidify DRO samples.**

7.6.7 Plug a long-stem glass funnel with a little soxhlet-extracted glass wool. Fill the funnel half-full with heat-purified granular sodium sulfate and place on a 250 mL collecting flask (500 mLs for BNA). Be sure each separatory funnel and corresponding collecting flask is appropriately labeled with the correct sample number, extraction batch number, and method code.

7.6.8 Seal and shake the separatory funnel vigorously for 2-minutes with periodic venting to release excess pressure. If emulsion occurs, the DCM can be separated by using the centrifuge (see **7.6.9.2 Centrifugation**).

CAUTION: Pressure builds up quickly in a separatory funnel that has both the stopcock closed and the stopper in place. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Be sure to vent the separatory funnel frequently by inverting the funnel and opening the stopcock. Do not point the funnel at or near anyone as it is being vented.

7.6.9 Emulsions

Allow the organic layer to separate from the water 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 one or more of the following techniques to complete the phase separation (document the presence of severe emulsions in the log book). Method 3510C, Section 7.6 specifies that samples that do not meet the 80% recovery of $MeCl_2$ after extraction should be re-extracted via Method 3520. The Project Manager will be contacted if this scenario occurs to determine the best course of action. Projects with samples that historically do not meet this requirement need to be logged with the 3520 preparation method to ensure that sufficient sample volume is available and holding times are met.

7.6.9.1 Stirring Rod

Physically break up the emulsion by stirring.

7.6.9.2 Centrifugation

Centrifugation separates liquids by density. Draw off the emulsion into a 125 mL, Teflon, centrifuge bottle. Balance the centrifuge. Centrifuge for 2-5 minutes. Carefully pour the contents back into the separatory funnel and drain off the DCM layer. Alternatively, after centrifugation, draw out the DCM (bottom layer) directly from the centrifuge bottle using a disposable, 10 mL pipet and deliver it to the collecting flask. Return any aqueous phase back to the appropriate separatory funnel. Add the next DCM aliquot first to the centrifuge bottle as a rinse and then to the appropriate separatory funnel.

7.6.9.3 Salting Out

Addition of salt makes the aqueous phase too polar to support a less polar phase. Add ~100 to 200 g of heat-purified sodium chloride to the aqueous phase and shake the sample again.

7.6.10 Drain the organic layer through the funnel containing sodium sulfate and into the 250 mL collecting flask.

7.6.11 Repeat the extraction 2 more times using 60 mLs (30 mLs for LVI) of DCM each time, combining all 3 organic layers in the same flask. Rinse down the sodium sulfate with ~20 mLs of DCM after the first and last drain. For LVI, rinse the sodium sulfate after each shake. Unless BNA, the sample is now ready for concentration. Proceed to Section 7.6.12 for K-D concentration instructions

BNA Samples The samples are adjusted to pH>11 and extracted 3-more times as described above, after which, the samples are ready for concentration. Proceed to Section 7.6.12 for K-D concentration instructions.

7.6.12 K-D Sample Concentration

Occasionally, samples extracted from wastewaters for subsequent BNA analysis and/or Pesticide/PCB analysis receive a gel permeation cleanup (GPC). These samples need to be identified prior to concentrating. Refer to the appropriate section of USP-001 or 003 for the concentration of these "pre-GPC" BNA samples. Refer to the appropriate section of USP-003 for the concentration of these "pre-GPC" Pesticide/PCB samples.

7.6.12.1 Assemble a K-D concentrator by attaching a 10 mL concentrator tube (insulated type if BNA or DRO) to a 500 mL K-D flask. Add a couple of boiling chips. Transfer each extract to its own K-D flask (making sure that each flask is appropriately labeled). Rinse the Erlenmeyer flask 3 times with DCM, adding each rinse to the K-D flask. Rinse down the joint with DCM and then attach a 3-ball Snyder column to each and place on the water bath. The temperature control should be set at medium. This setting should correspond to 80-90°C. Concentration to <10 mLs is usually completed in 60-90 minutes. The balls should chatter but the chambers should not flood.

7.6.12.2 Refer to appropriate test for further concentration instructions.

BNA and DRO When the apparent final volume reaches about 2-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs DCM. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with DCM into the receiver. Cover the receiver with a piece of aluminum foil. If the extracts are to sit overnight, they are to be stored at 4 ± 2°C.

Proceed to Section 7.6.13 for further concentration using nitrogen evaporation.

PCB and Pesticide/PCB When the apparent volume reaches ~ 5 mLs, a solvent exchange is required. Add 50 mLs of hexane to the K-D flask (through the Snyder column). Concentrate the extract by raising the temperature of the water bath, if necessary, to maintain proper distillation. The Snyder column may be wrapped with aluminum foil to aid in proper evaporation. When the apparent volume reaches 3-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs hexane. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with hexane into the receiver.

Transfer the extract into a labeled, 16X125 test tube that has been previously marked at 10.0 mLs. Rinse the receiver 3 times with hexane, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

Proceed to Section 7.6.13 for further concentration using nitrogen evaporation.

PAHs When the apparent volume reaches ~5 mLs, a solvent exchange is required. Add 5 mLs of acetonitrile to the K-D flask (through the Snyder column). Concentrate the extract by raising the temperature of the water bath, if necessary, to maintain proper distillation. The Snyder column may be wrapped with aluminum foil to aid in proper evaporation. When the apparent volume reaches 3-5 mLs, remove from the bath. Allow to drain and cool for 10 minutes. Remove the Snyder column, rinsing the joint and flask with 1-2 mLs acetonitrile. Allow to drain. Dry off the outside of the joint between the flask and receiver to remove the water. Pull the 2 pieces apart gently, in a twisting motion. Rinse off the joint with acetonitrile into the receiver.

Transfer the extract into a labeled, 16X125 test tube that has been previously marked at 5.0 mLs. Rinse the receiver 3 times with acetonitrile, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

7.6.13 TurboVap Sample Concentration

All samples being concentrated must be recorded in the extraction log.

**Occasionally, samples extracted from soil/sediments for subsequent BNA analysis and/or Pesticide/PCB analysis receive a gel permeation cleanup (GPC). These samples need to be identified prior to concentrating. Refer to UP-SP-003 for this procedure.

Prepare the TurboVap by ensuring that the unit has sufficient nitrogen supply and the water level in the bath is greater or equal to the sample volume in the TurboVap tube. The pressure should be set to between 8 to 15 PSI.

Transfer the extract to an appropriately labeled TurboVap tube. It can only contain 200 mLs of extract. If there is more than 200 mLs of extract to concentrate, concentrate 200 mLs of it down to approximately 20 mLs, then add the remaining extract to the tube. Rinse the Erlenmeyer flask 3 times with DCM, adding each rinse to the tube.

To begin the concentration process, gently place the tube in the water bath. Close the cover. The TurboVap has several endpoint selections. Select the sensor endpoint. The following parameters should be set: bath temperature to 40, endpoint time to 00, and pressure to 14 PSI. Press the START/STOP button for each cell position used. To pause the concentration, raise the cover. To continue operation, lower the cover. When a cell reaches its endpoint, its corresponding light blinks and the beeper sounds briefly every thirty seconds. Raise the cover to silence the beeper. Remove the glassware from the bath.

Refer to the appropriate 'parameter' defined below for further concentration instructions:

BNAs

At the endpoint the extract should be at ~1 mLs. The extract should be removed from the TurboVap before the endpoint sensor is triggered. Low analyte recovery will occur if the extract volume is concentrated to low. Transfer the entire extract to an appropriately labeled 2 mL vial. Rinse the TurboVap tube with a few 100 uLs of DCM 3 times, transferring the rinse to the 2 mL vial each time.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

PCB, Pesticide/PCB

Begin concentrating extract. When an extract volume reaches 10 mLs, perform a hexane exchange. Remove the tube from the TurboVap and add 50 mLs of hexane. This is done to ensure that solvent is not spilled in the water bath. Place the tube back on the TurboVap and concentrate the extract back down to 10 mLs. Remove the glassware from the bath.

Transfer the extract into a labeled, 16 X125 test tube that has been previously marked at a 10.0 mL* (5.0 mL for LVI) volume. Rinse the TurboVap tube 3 times with hexane, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

* Volumetrically pipet 10.0 mL (5.0 mL for LVI) of hexane into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

PAHs

Before adding the extract to the TurboVap tube, add 5 mLs of acetonitrile.

Concentrate the extract to 10 mLs. Transfer the extract into a labeled, 16 X125 test tube that has been previously marked at a 5.0 mL* volume. Rinse the TurboVap tube 3 times with acetonitrile, transferring the rinsate each time to the 16X125 test tube. Refrigerate the extracts if they are not going to be immediately concentrated.

* Volumetrically pipet 5.0 mL of DCM into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

DRO

At the endpoint the extract should be at approximately 10 mLs. Transfer the extract into a 16 X 125 test tube that has been previously marked at a 5.0 mL* (2.5 mL for LVI) volume. Rinse the TurboVap tube 3 times with DCM, transferring the rinsate each time to the 16 X 125 test tube.

* Volumetrically pipet 5.0 mL (2.5 mL for LVI) of DCM into a 16 X 125 test tube. Mark the meniscus using a fine-tipped marker. Discard the solvent into an appropriate waste vessel.

Proceed to Section 7.6.14 for further concentration using nitrogen evaporation.

7.6.14 Nitrogen Evaporation

7.6.14.1 For further concentration, place each receiver or test tube on the N-Evap tray. The N-Evap outlet needles should be stored in a 600 mL beaker containing DCM:Acetone (1:1) mixture prior to each use. Wipe each outlet needle with a KimWipe and lock into place on the N-Evap. Lower the outlet tube into the receiver being careful not to touch the sides of the receiver. The outlet tube should remain above the surface of the extract. Open the valve of the nitrogen tank and adjust the pressure in order to achieve the appropriate nitrogen stream. The nitrogen stream should make a slight "dimple" in the surface of the extract. If the extract bubbles, the nitrogen stream is **not** gentle enough. Lower the entire N-Evap tray into the warm water bath to aid the evaporation process. Heat is not necessary and if used the temperature should not exceed 35°C.

7.6.14.2 The extracts must never be allowed to go dry. While the extracts are concentrating, occasionally rinse down the sides of the receiver or test tube with appropriate solvent (see corresponding test instructions below).

7.6.14.3 Refer to appropriate test below for further concentration instructions.

BNA and 625

7.6.14.5a Once the extracts reach ~0.5 mLs, remove from the N-Evap. Label a 1.5-2 mL screw capped vial with appropriate sample number and batch number. Transfer the 0.5 mLs to the vial using a transfer pipet. Rinse the receiver well with a little (<1/4 mL) DCM and add to the vial. Compare the sample volume in the vial to a pre-marked, 0.9 mL vial. Rinse the receiver with a little more DCM (<1/4 mL). Transfer to sample vial. Compare the volume to the pre-marked 0.9 mL vial. Dilute the sample (if necessary) to 0.9 mLs with another DCM rinsate. The extraction analyst must rinse the receiver at least 2-times and stay at or below 0.9 mLs. The use of a 500 uL syringe gives the analyst more control when adding the small-volume DCM rinses to the receiver. If the rinsates make the extract more than the 0.9 mL, the analyst may hold the vial under the stream of nitrogen momentarily to reduce the volume back down to 0.9 mL. The GC/MS analyst will bring the sample extract to exactly 1.0 mL prior to analysis. Seal vial with a Teflon-lined screw-cap.

There will be samples that will not concentrate to a 1 mL final volume. In these cases, bring the sample up to the smallest final volume (increments of 1 mL)* that the sample and 3 rinses will transfer into. All changes in final volume must be documented LIMS. Notify your supervisor.

* Volumetrically add the appropriate volume of DCM to a 16 X 125 screw top test tube and mark the meniscus using a fine-tipped marker. Discard the DCM into an appropriate waste vessel. Bring up sample to volume using this test tube.

7.6.14.6a All samples pertaining to one extraction batch are placed in a plastic vial holder and secured. A listing of the entire contents of the batch, including clients, sample numbers, batch number, standard numbers, and date extracted, is to be placed WITH the set. To relinquish samples, the extraction analyst takes the sample extracts to the sample extract refrigerator (located in the GC/MS Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location.

7.6.14.7a An analyst from the GC/MS BNA group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

PCBs, Pesticide/PCB and 608

7.6.14.4b Concentrate the sample to 2-3 mLs and dilute to the 10.0 mL (5.0 mL for LVI) mark with hexane.

PCBs Refer to mandatory sulfur acid cleanup (Sec. 7.6.16)

7.6.14.5b All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample number, and test code written on it.

7.6.14.6b To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the GC Extractables group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

PAHs

7.6.14.4c Concentrate the sample to ~2 mLs to remove any remaining DCM. Dilute the sample up to 5.0 mLs with acetonitrile.

7.6.14.5c All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample number, and test code written on it.

7.6.14.6c To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables Lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the HPLC group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

Diesel/DRO

7.6.14.4d Concentrate the sample to the 5.0 mL (2.5 mL for LVI) mark. If the solvent goes below the 5.0 mL (2.5 mL for LVI) mark, dilute to 5.0 mLs (2.5 mL for LVI) with DCM.

7.6.14.5d All samples pertaining to one extraction batch are placed in a test tube rack. Each test tube should have the extraction batch number, sample number, and test code written on it.

7.6.14.6d To relinquish samples, the extraction analyst takes the sample extracts to the sample cooler area (located in the GC Extractables lab). The TALS ICOC module is used by the prep analyst to electronically transfer custody of the extracts to the storage location. An analyst from the GC Extractables group must be present to receive the samples. Both analysts check for completeness and correctness of the information. All extracts are to be kept at 4 ± 2°C in the dark, until analysis.

7.6.15 Screening and Cleanup

7.6.15.1 Samples are not pre-screened prior to extraction. Unless previous experience has proven high concentrations and/or background, a 1-liter sample will be extracted.

7.6.15.2 Sludge samples are a mixture of water and suspended solid materials. If the percent moisture of a given sludge is > 90%, a 10 g or 30 g portion may be weighed and added to 1-L of reagent water. It is then extracted like a water matrix as described in this procedure. Despite the extraction method, sludges are generally reported on a dry weight basis and the total solids result must be entered into the spreadsheet.

7.6.15.3 PAH samples do not receive any cleanup procedures.

7.6.16 Extract Cleanup by Gel Permeation Chromatography (GPC)

7.6.16.1 It is not mandatory that BNA or Pesticide/PCB samples be subjected to further cleanup steps prior to analysis. However, if it is requested that these sample sets receive a GPC cleanup, refer to the appropriate section of the UP-SP-003.

7.6.16.2 It is not mandatory that Pesticide/PCB samples be subjected to further cleanup steps prior to analysis. However, if it is requested that the Pesticide/PCB set receives a Florisil cleanup, refer to the appropriate sections of UP-SP-003.

7.6.16.3 It is not mandatory that DRO sample be subjected to further cleanup procedures prior to analysis. However, if it is requested that the DRO set receives a Silica Gel cleanup, refer to the appropriate section in UP-SP-003.

7.6.17 Mandatory Acid Cleanup for PCBs

If the sample is to be analyzed for **PCBs ONLY**, a mandatory sulfuric acid cleanup is performed. Add ~3-4 mLs of concentrated H_2SO_4 to the extract (contained in a screw-top test tube). Shake vigorously for a least 1 minute. Centrifuge the hexane/acid for at least a couple minutes to ensure adequate separation. If the acid layer (the bottom layer) is dark in color, transfer the hexane layer (the top layer) to another screw-top test tube and repeat the acid cleanup. The acid cleanup should be repeated until the acid layer no longer turns dark in color. The hexane extract must always be immediately removed from the acid and transferred into another clean, labeled, screw-top test tube. Document the acid cleanup in LIMS. Relinquish the samples as described in the nitrogen evaporation section.

7.7 Documentation

7.7.1 All extraction information must be carefully documented in 'real time' in the LIMS batch and prep worksheet (Attachment 1). Any additional sample or extraction information is recorded in the comment section of the batch sheet. All problems and/or deviations from normal procedures must be documented in LIMS. The supervisor should be notified and a Non-Conformance Memo (NCM) may need to be initiated.

7.7.2 Different parameters are given different test methods within LIMS. Refer to the batch paperwork for the assigned test methods.

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 Method Blank (MB)

The MB is reagent (Milli-Q) water, which has been spiked with the appropriate surrogate, then taken through the entire extraction procedure and is used to monitor the introduction of artifacts into the process. This demonstrates that the materials used are free of interferences.

8.1.2 Laboratory Control Sample (LCS)

The LCS is reagent (Milli-Q) water, which has been spiked with a predetermined quantity of spike solution with the appropriate analytes of interest and the appropriate surrogate.

8.1.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

In MS/MSD analysis, predetermined quantities of spike solutions of specified analytes and appropriate surrogate are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected by the analysis. The relative percent difference between the samples is calculated and used to assess analytical precision.

8.1.4 Surrogate

A surrogate is an organic compound(s) which is similar to the analytes of interest in chemical composition, extraction and chromatography, but which is not normally found in environmental samples. The surrogate is added to **all** samples, MBs, MS/MSD and LCS prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

8.1.5 Sometimes PCBs are extracted in the same extraction batch as Pesticide samples. When this occurs, the QC includes one MB; one LCS/LCSD for the pesticide compounds; one LCS/LCSD for PCB compounds; one MS/MSD for the pesticide compounds; and one MS/MSD for PCB compounds.

8.1.6 LCDs and MSDs are not performed for BNA TCLP.

8.1.7 LCSD and MSDs are not performed for Pesticide TCLP samples. However, an LCS/LCS along with a MS/MS are extracted and recorded. The TCLP Pesticide Spike and Toxaphene Spike need to remain separate from one another.

8.2 Corrective Action

The extraction analyst must perform the Quality Control described in Section 7.1. The extraction analyst will not know if all QC is in control until the GC/MS, GC/Extractables, or HPLC group analyzes the extracts and determine the results of the MBs, MS/MSDs, LCSs, and surrogate recoveries. It is the responsibility of the analysis group to inform the Organic Extractions Department by documenting in the "BNA Re-Extraction Log" or by initiating a Non-Conformance Memo (NCM) in LIMs when a sample or set of samples requires re-extraction. Re-extractions are to be designated as R1 in the extraction process.

All problems and/or deviations from normal procedures must be documented in LIMS. Your supervisor should be notified and a NCM may need to be initiated.

9.0 DATA ANALYSIS AND CALCULATIONS

Not Applicable.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Waste flammable solvents. Solvents will be collected in a waste jar and poured into the Waste Chlorinated Solvent drum using a funnel to reduce splashing.
- Extracted water samples will be poured into the Waste Water drum using a funnel to reduce splashing.
- Remaining solid waste produced by this method shall be disposed of into the Non-Hazardous Waste solids containers.
- Any unused extracts remaining from this method shall be turned over to the EHSC to be collected for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Section 1, 7 and 8.

12.0 REFERENCES

Refer to Sections 1, 6, 7 and 8.

13.0 ATTACHMENTS

Attachment 1. Example: Extraction Prep Worksheet
Attachment 2. Example: Procedural Flowcharts
Attachment 3: Example: BNA Surrogate Stock Solution
Attachment 4: Example: 8270 LCS Mix
Attachment 5: Example: 3,3'-Dimethylbenzidine

Appendix 1: BNA Halowax
Appendix 2: Alachlor/Atrazine
Appendix 3: Wisconsin DRO
Appendix 4: JP4/JP8
Appendix 5: AR1242/AR1254

14.0 REVISION HISTORY

- Revision 13, was updated on 02/20/13
- Update for additional LVI procedures
- Update Section 7.6.2 to include the gravimetric determination of initial volume.

Attachment 1.

Example: Extraction Prep Worksheet (027-001 to 027-007)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-178198 | Analyst: Heinrich, Stephanie C | Batch Open: 2/21/2013 8:49:17AM

Method Code: 500-3510C-500 | Batch End: 2/21/2013 9:49:00AM

Liquid-Liquid Extraction (Separatory Funnel)

Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	GrossWt TareWt	InitAmnt FinAmnt	PHs Rcvd	PHs Adj1	PHs Adj2	Due Date	Analytical TAT	Div Rank	Comments
MB 500-178198/1	MB~500-178198/1 N/A	N/A		1000 mL / 1.0 mL	6	2	12	N/A	N/A	N/A	
LCS 500-178198/2	LCS~500-178198/2 N/A	N/A		1000 mL / 1.0 mL	6	2	12	N/A	N/A	N/A	
LB 500-178066/1-A	LB~500-178066/1-A N/A	N/A		100 mL / 1.0 mL	6	2	12	N/A	N/A	N/A	
LB2 500-178146/1-A	LB2~500-178146/1-A N/A	N/A		100 mL / 1.0 mL	6	2	12	N/A	N/A	N/A	
500-54671-A-1-A	500-54671-A-1-A (8270D)	N/A		100 mL / 1.0 mL	6	2	12	2/25/13	4_Days - E	3	
500-54671-A-1-A MS	500-54671-A-1-A~MS (8270D)	N/A		100 mL / 1.0 mL	6	2	12	2/25/13	4_Days - E	3	
500-54699-A-1-B	500-54699-A-1-B (8270D)	N/A		100 mL / 1.0 mL	6	2	12	2/25/13	3_Days - R	2	

(027-001)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-178198 | Analyst: Heinrich, Stephanie C | Batch Open: 2/21/2013 8:49:17AM

Method Code: 500-3510C-500 | Batch End: 2/21/2013 9:49:00AM

Batch Notes

Person's name who did the prep	SH
Prep Solvent Name	DCM
Prep Solvent Lot #	2080741
Prep Solvent Volume Used	360
Person's name who witnessed reagent drop	
Acid used for pH adjustment	sulfuric acid
Acid used for pH adjust Lot #	2010484
Base used for pH adjustment	sodium hydroxide
Base used for pH adjust Lot #	2072117
Silica Gel Lot Number	
Person's name who did the concentration	JD
Exchange Solvent Name	
Exchange Solvent Lot #	
Concentration Start Time	0950
Concentration End Time	1130
Final Concentrator Volume	1.0
Na2SO4 Lot Number	2067572
ID number of the thermometer	
Uncorrected Temperature	
Oven, Bath or Block Temperature 1	

(027-002)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-178198 | Analyst: Heinrich, Stephanie C | Batch Open: 2/21/2013 8:49:17AM

Method Code: 500-3510C-500 | Batch End: 2/21/2013 9:49:00AM

N-evap #	C-0655
N-evap temperature	29.6
Uncorrected N-evap Temperature	30.0
Sufficient volume for MS/MSD?	Y
Florisil Lot #	
TBA Lot #	
Copper Lot #	
Acid used for Clean Up Reagent	
Pipette ID	
Syringe Lot #	A26,B23
pH Paper Lot Number	
Filter Paper Lot Number	
Glass Wool ID	2067572
NaCL Lot #	
Sodium Bicarbonate ID	
Batch Comment	

Comments

Login Comments for Job 54671: Form 8 required for all report levels.
Internal COC
BP LaMP protocols

(027-063)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-177529 | Analyst: Knieriemen, Dan A | Batch Open: 2/13/2013 1:34:48PM

Method Code: 500-3510C_LVI-500 | Batch End: 2/13/2013 2:30:00PM

Liquid-Liquid Extraction (Separatory Funnel)

	Input Sample Lab ID	Input Sample Lab ID (Analytical Method)	SDG	GrossWt TareWt	InitAmnt FinAmnt	PHs Rcvd	PHs Adj1	PHs Adj2	Due Date	Analytical TAT	Div Rank	Comments
1	MB 500-177529/1	MB~500-177529/1 N/A	N/A		125 mL / 5.0 mL	6			N/A	N/A	N/A	
2	500-54465-A-1	500-54465-A-1 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
3	500-54465-A-2	500-54465-A-2 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
4	500-54465-A-3	500-54465-A-3 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
5	500-54465-A-4	500-54465-A-4 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
6	500-54465-A-5	500-54465-A-5 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
7	500-54465-A-6	500-54465-A-6 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
8	500-54465-A-7	500-54465-A-7 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
9	500-54465-A-8	500-54465-A-8 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	
10	500-54465-A-9	500-54465-A-9 (8081B)	N/A		125 mL / 5.0 mL	6			2/19/13	6_Days - R	4	

(024-004)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-177529 Analyst: Knieriemen, Dan A Batch Open: 2/13/2013 1:34:48PM

Method Code: 500-3510C_LVI-500 Batch End: 2/13/2013 2:30:00PM

11	500-54465-A-10	500-54465-A-10 (8081B)	N/A		125 mL	6			2/19/13	6_Days - R	4	
					5.0 mL							
12	500-54465-A-11	500-54465-A-11 (8081B)	N/A		125 mL	6			2/19/13	6_Days - R	4	
					5.0 mL							
13	500-54465-A-12	500-54465-A-12 (8081B)	N/A		125 mL	6			2/19/13	6_Days - R	4	
					5.0 mL							
14	500-54465-A-13	500-54465-A-13 (8081B)	N/A		125 mL	6			2/19/13	6_Days - R	4	
					5.0 mL							

(027-005)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-177529 | Analyst: Knieriemen, Dan A | Batch Open: 2/13/2013 1:34:48PM

Method Code: 500-3510C_LVI-500 | Batch End: 2/13/2013 2:30:00PM

Batch Notes

Person's name who did the prep	dak
Prep Solvent Name	DCM
Prep Solvent Lot #	2080741
Prep Solvent Volume Used	
Person's name who witnessed reagent drop	
Acid used for pH adjustment	
Acid used for pH adjust Lot #	
Base used for pH adjustment	
Base used for pH adjust Lot #	
Silica Gel Lot Number	
Person's name who did the concentration	LK
Exchange Solvent Name	Hexane
Exchange Solvent Lot #	2045695
Concentration Start Time	1800
Concentration End Time	2030
Final Concentrator Volume	5.0
Na2SO4 Lot Number	2070305
ID number of the thermometer	
Uncorrected Temperature	
Oven, Bath or Block Temperature 1	

(027-006)

Aqueous Extraction Prep Worksheet

Used for Collecting Prep Info)

Batch Number: 500-177529 | Analyst: Knieriemen, Dan A | Batch Open: 2/13/2013 1:34:48PM

Method Code: 500-3510C_LVI-500 | Batch End: 2/13/2013 2:30:00PM

N-evap #	C-2091
N-evap temperature	29.9
Uncorrected N-evap Temperature	30
Sufficient volume for MS/MSD?	n
Florisil Lot #	
TBA Lot #	
Copper Lot #	
Acid used for Clean Up Reagent	
Pipette ID	
Syringe Lot #	a20, a22, a23
pH Paper Lot Number	
Filter Paper Lot Number	
Glass Wool ID	2067572
NaCL Lot #	2070341
Batch Comment	

Comments

Login Comments for Job 54465: MDL_8081B_W_LVI + DOCs

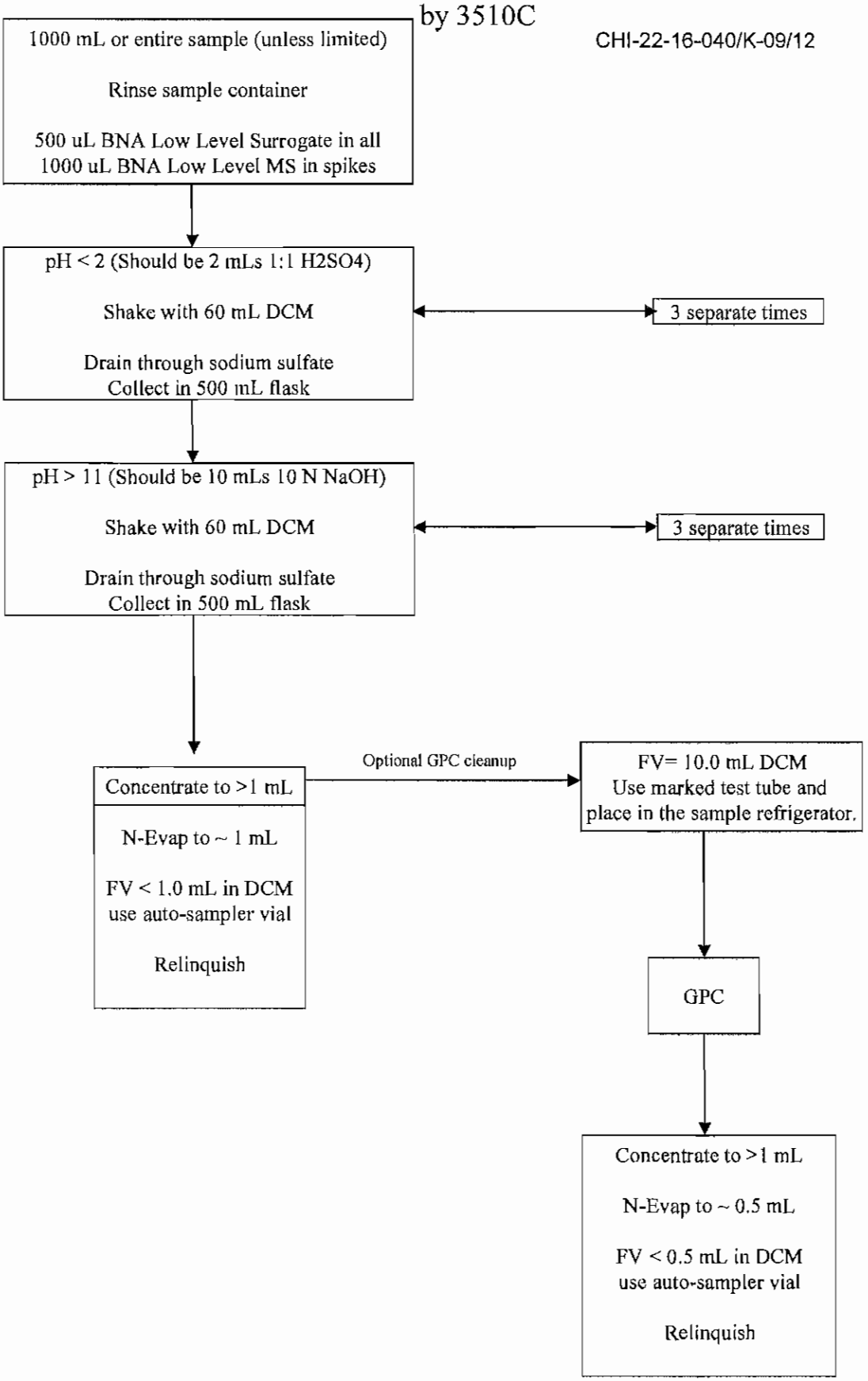
(027007)

Attachment 2.

Example: Procedural Flowcharts (028-001 to 028-016)

Flowchart for BNA Water
(8270C/8270D, 625)
by 3510C

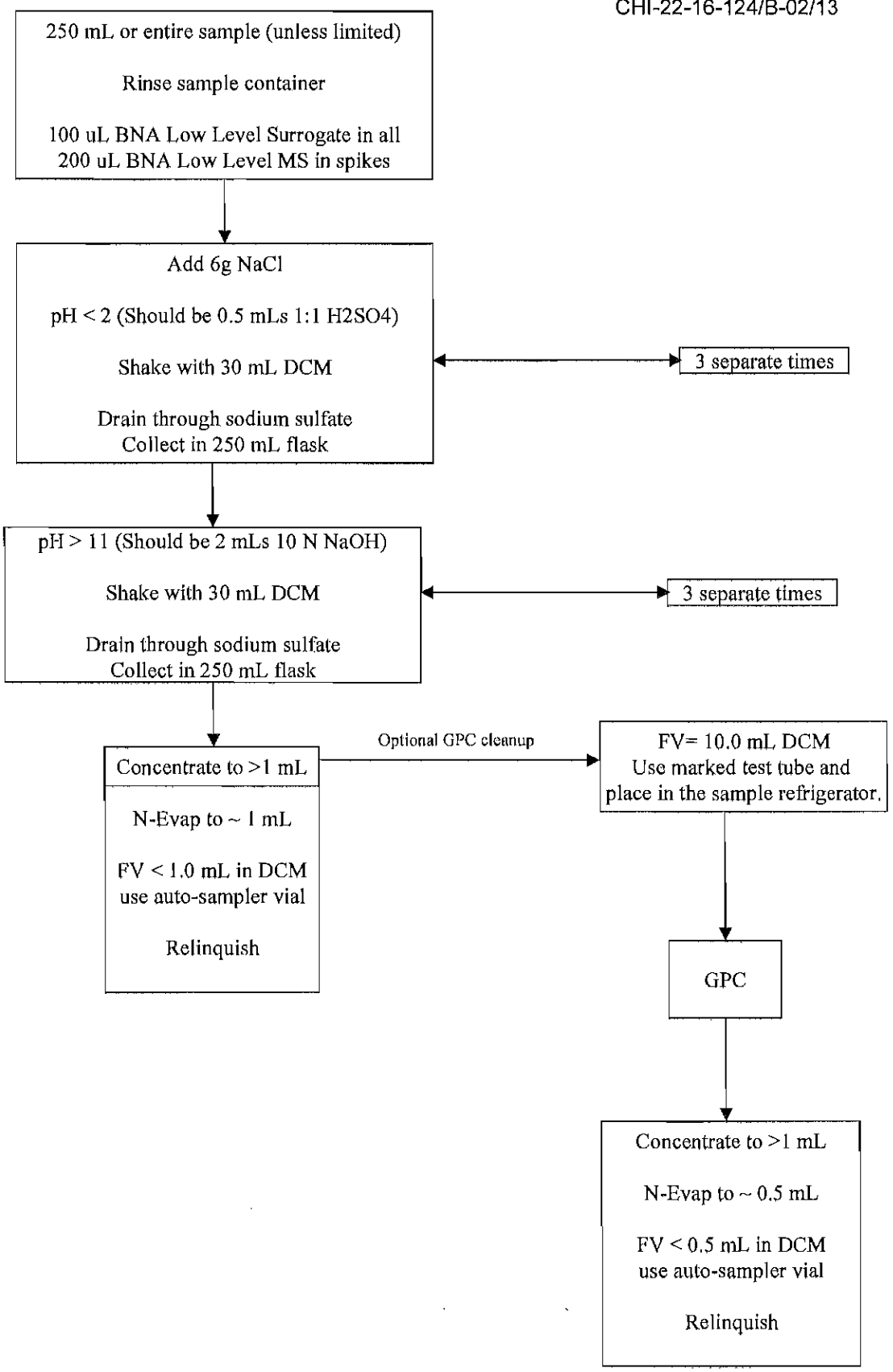
CHI-22-16-040/K-09/12



(028-001)

Flowchart for BNA Water LVI
(8270C/8270D, 625)
by 3510C_LVI

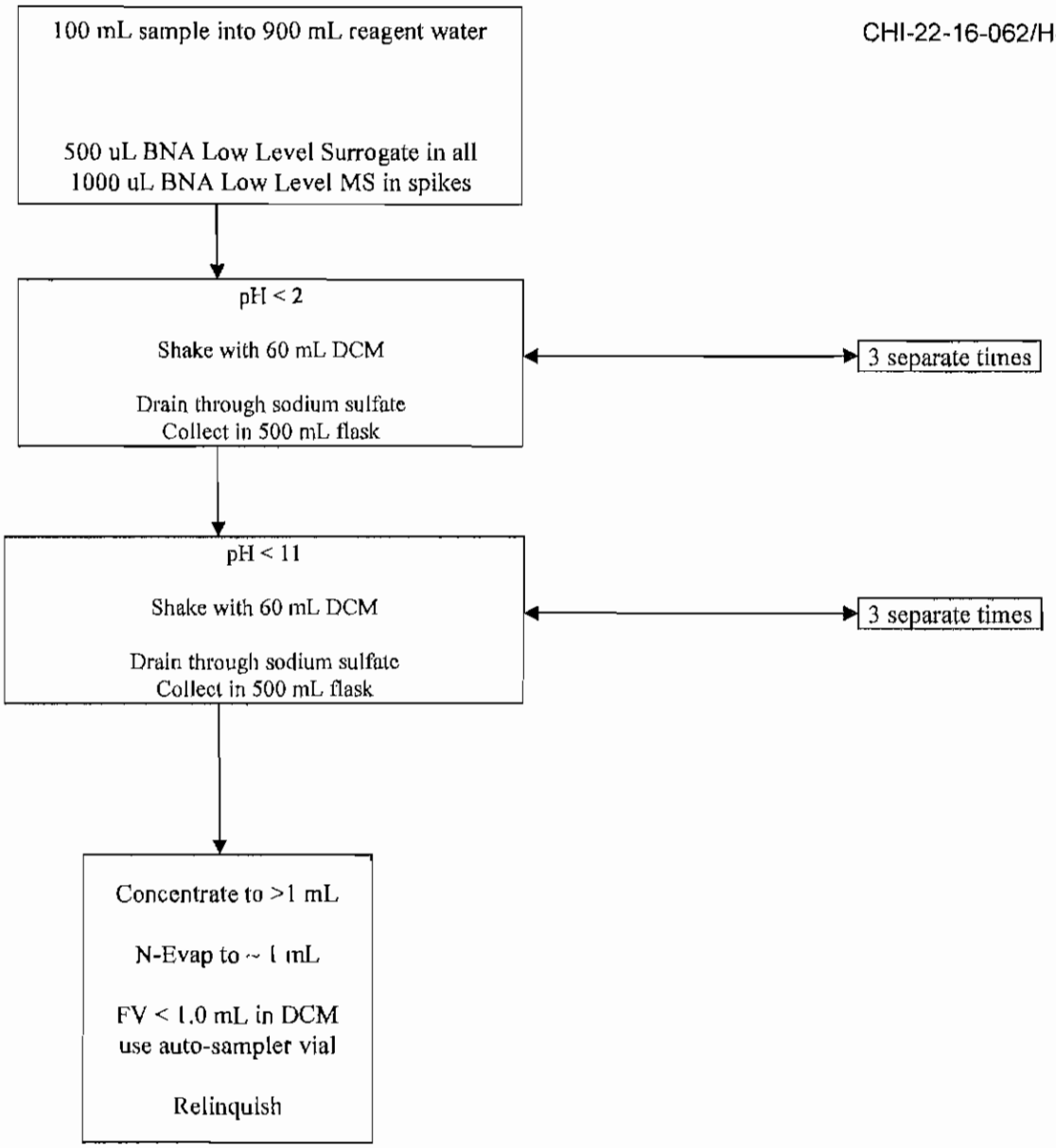
CHI-22-16-124/B-02/13



(028-002)

Flowchart for BNA TCLP
by 3510C

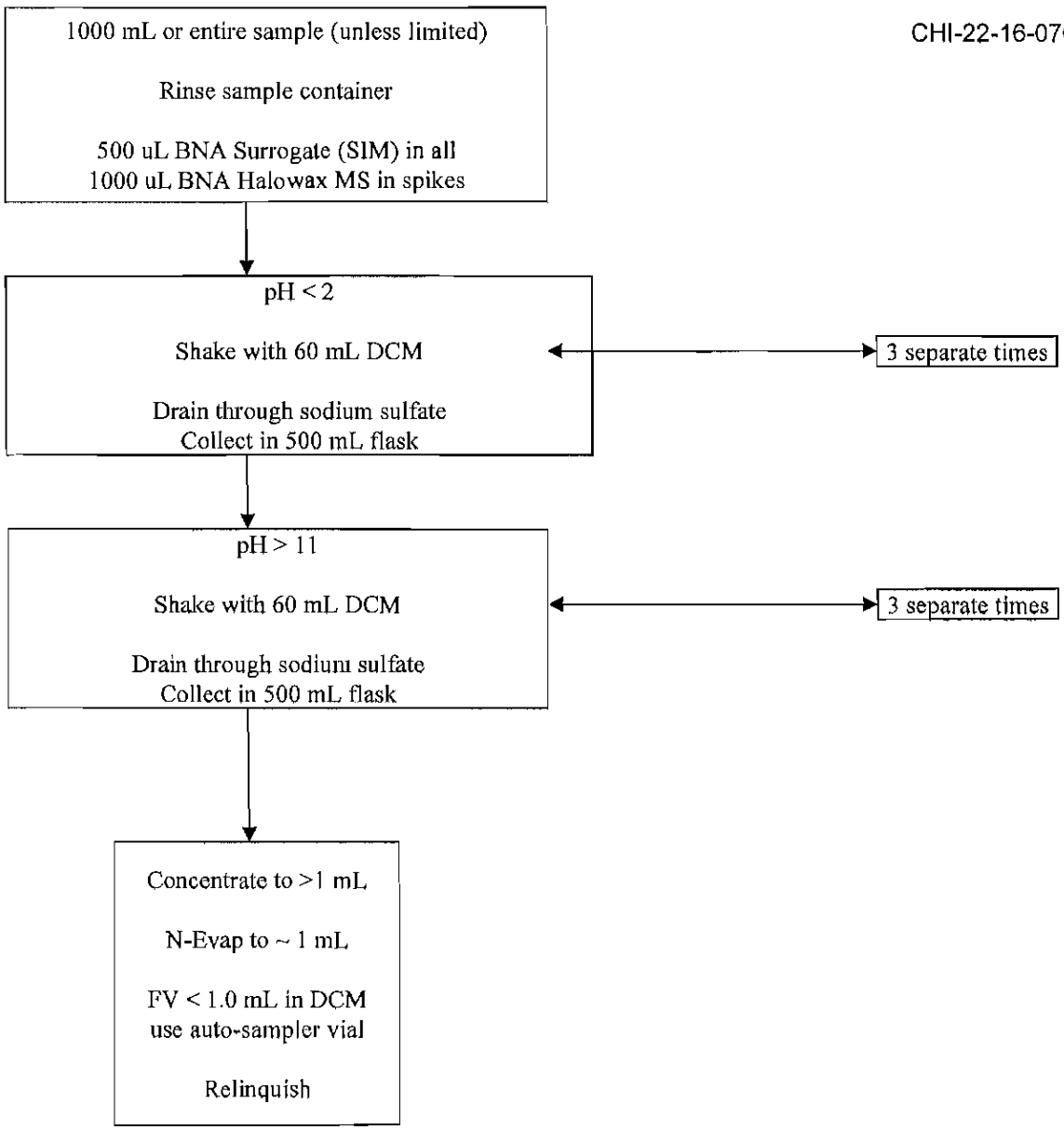
CHI-22-16-062/H-11/12



(028-003)

Flowchart for BNA Halowax Water
by 3510C

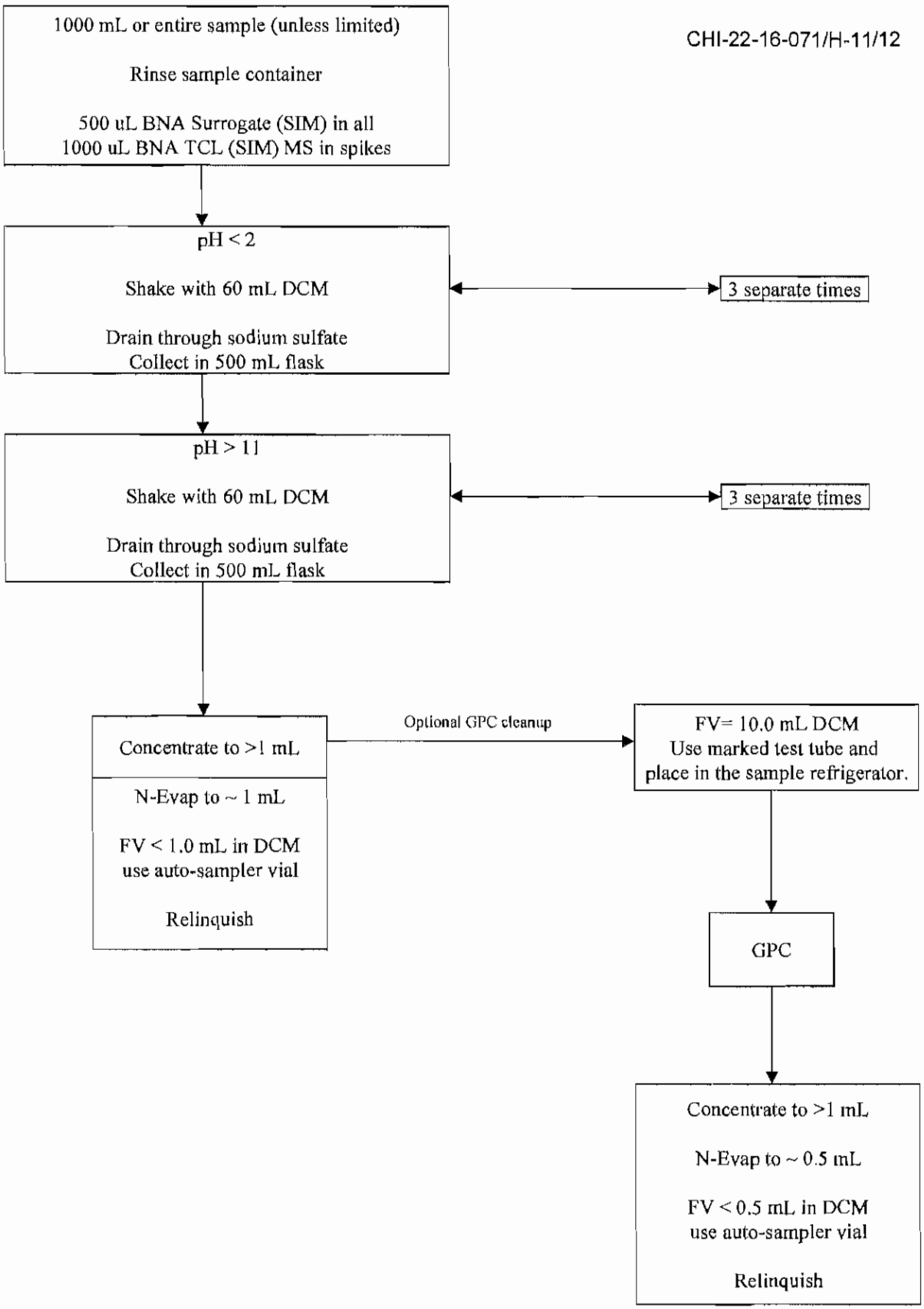
CHI-22-16-070/H-11/12



(028-004)

Flowchart for BNA SIM Water
by 3510C

CHI-22-16-071/H-11/12

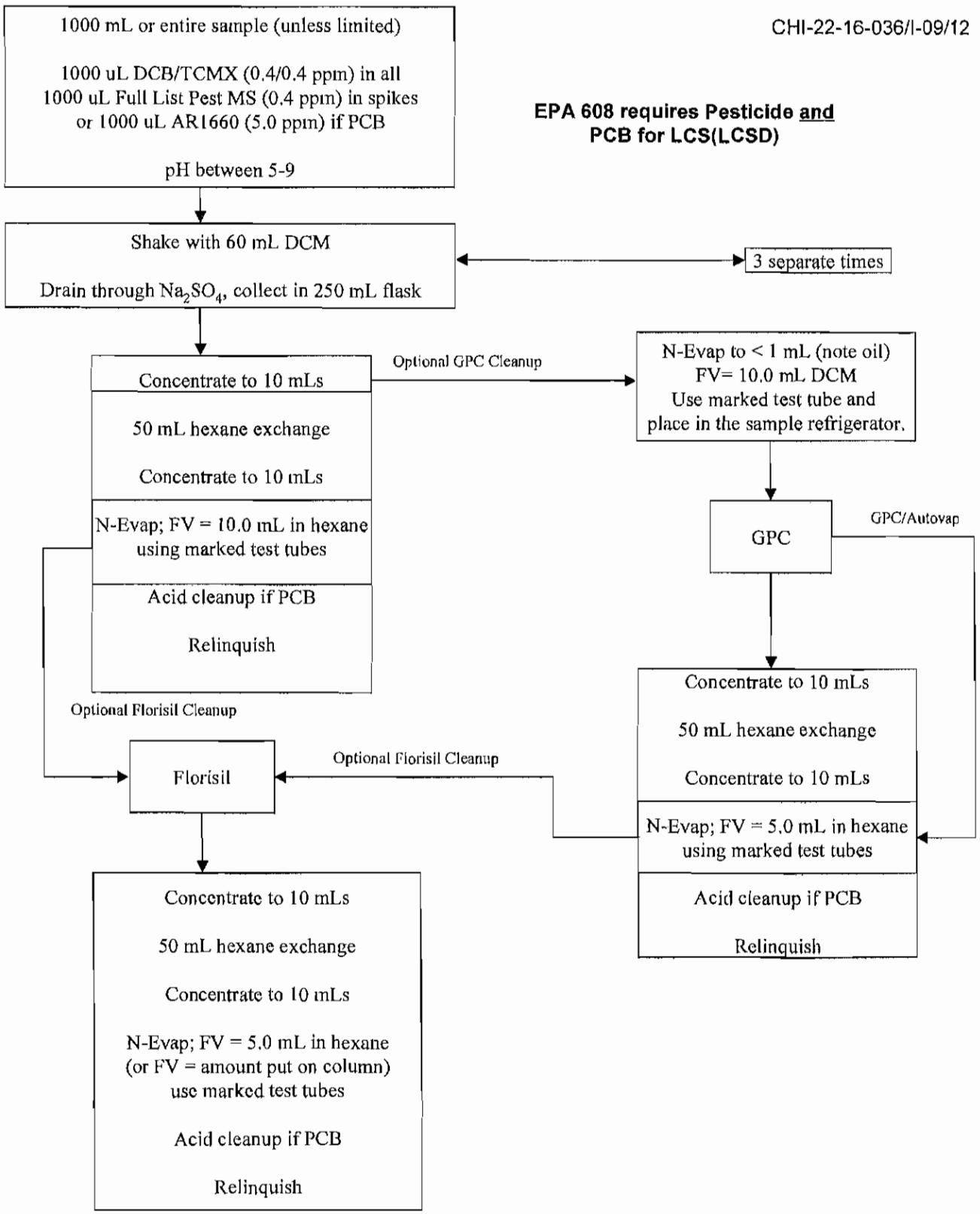


(028-005)

Flowchart for Pest/PCB Water (8081, 8082, 608) by 3510

CHI-22-16-036/I-09/12

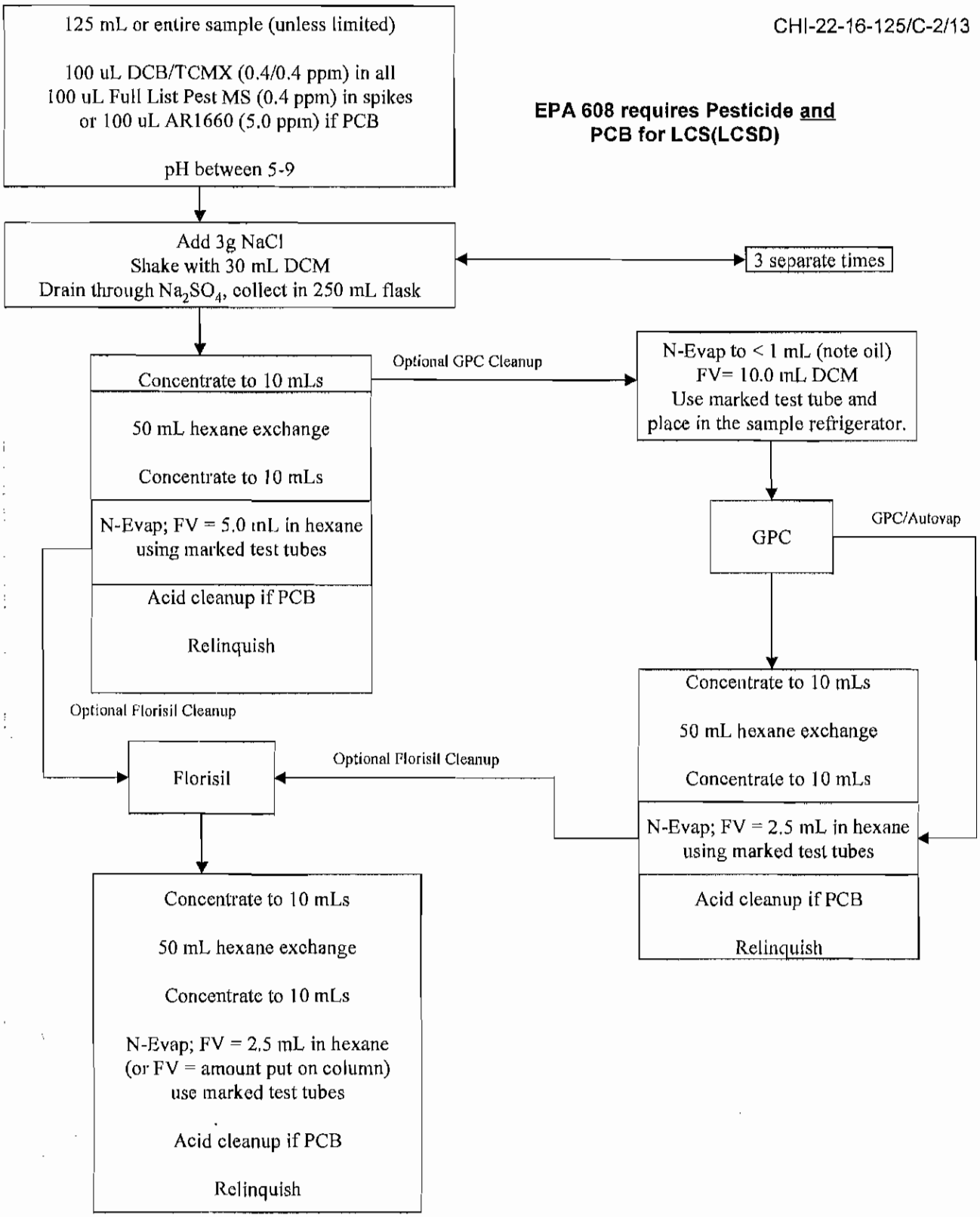
EPA 608 requires Pesticide and PCB for LCS(LCSD)



(028-006)

Flowchart for Pest/PCB LVI Water (8081, 8082, 608) by 3510_LVI

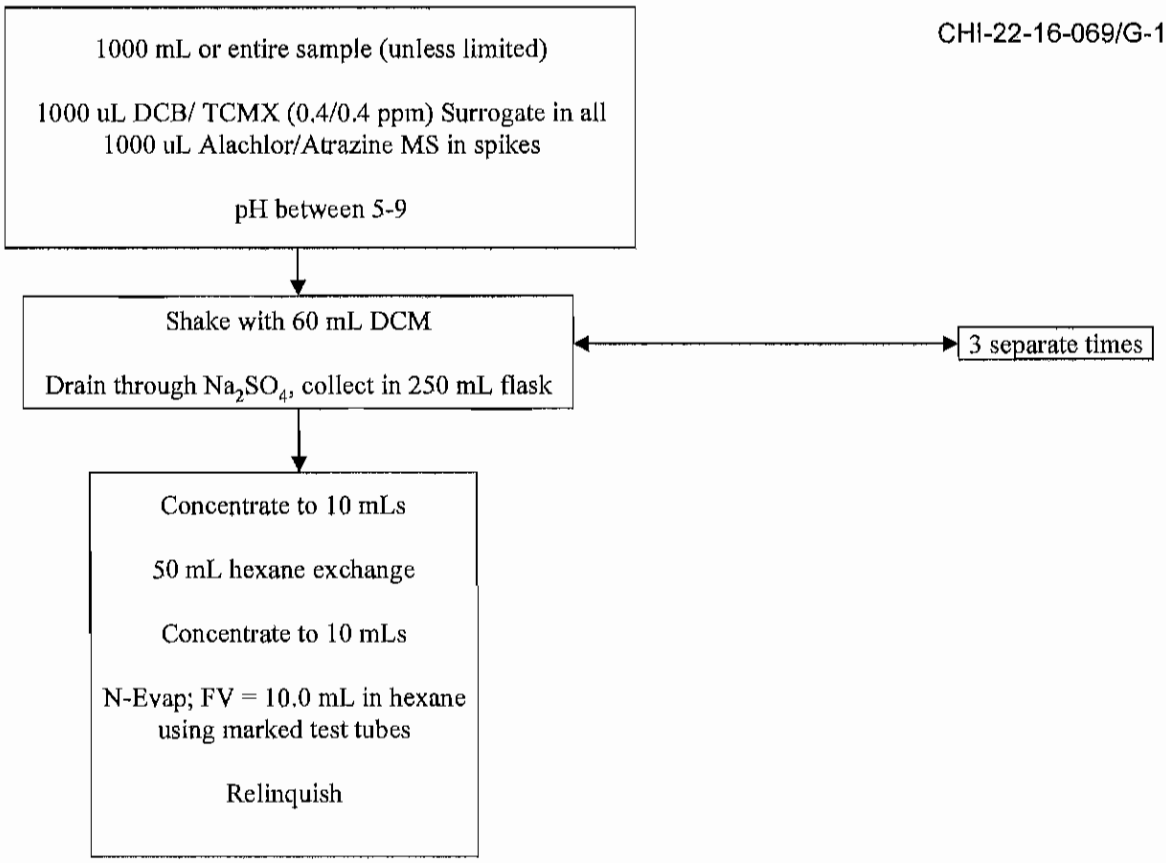
CHI-22-16-125/C-2/13



(028-007)

Flowchart for Alachlor/Atrazine Water (8081A) by 3510C

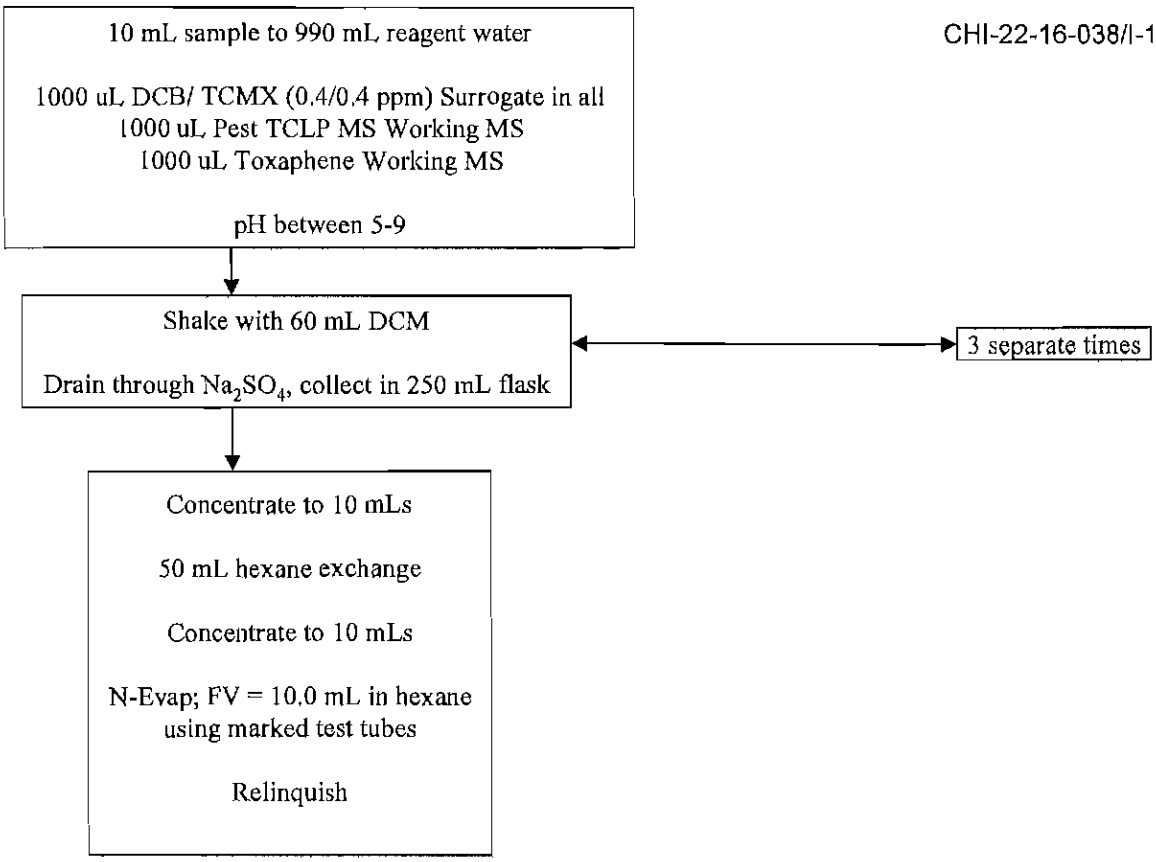
CHI-22-16-069/G-11/12



(D28-008)

Flowchart for Pest/PCB TCLP
by 3510C

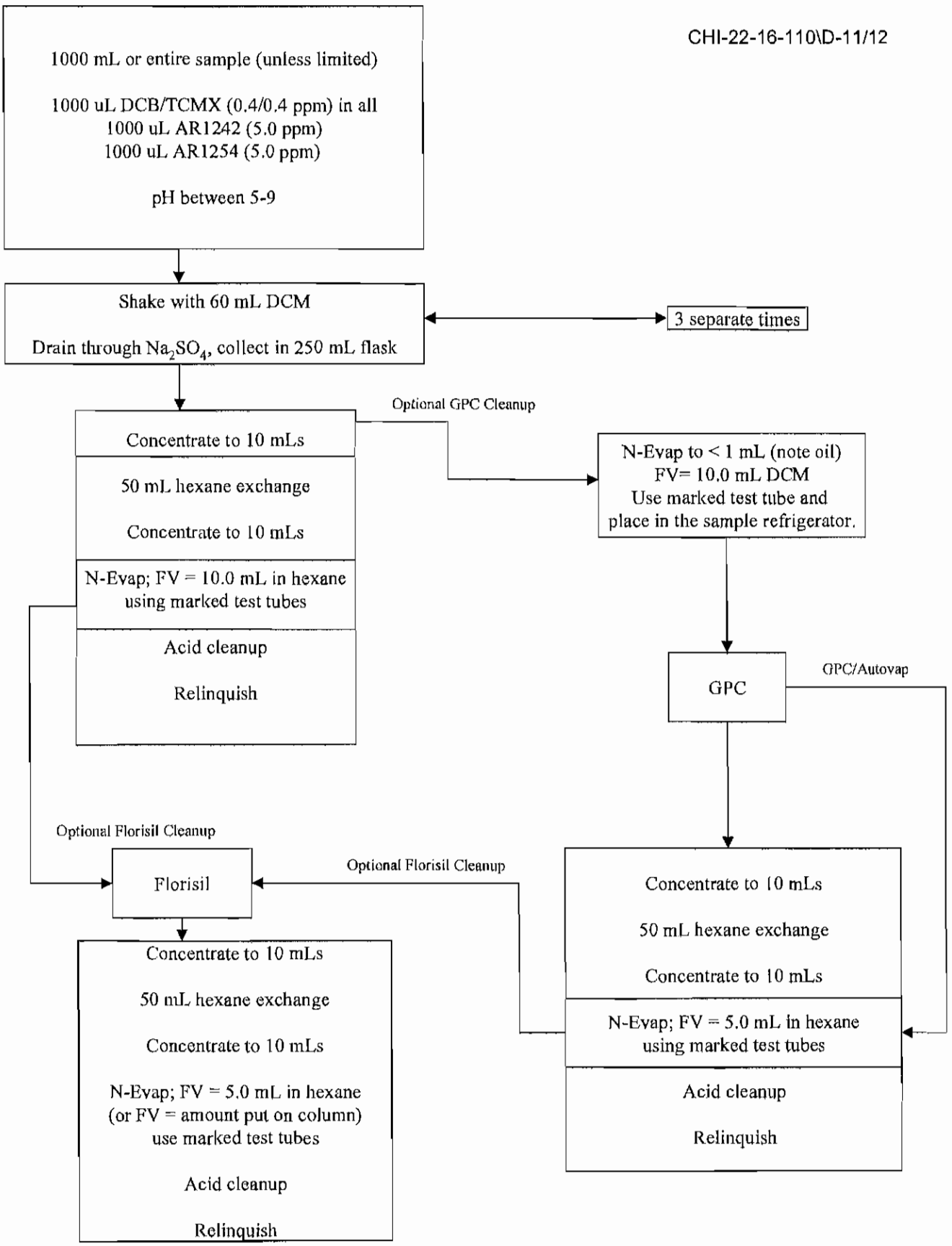
CHI-22-16-038/I-11/12



(028-009)

Flowchart for AR1242, AR1254 Water (8082) by 3510

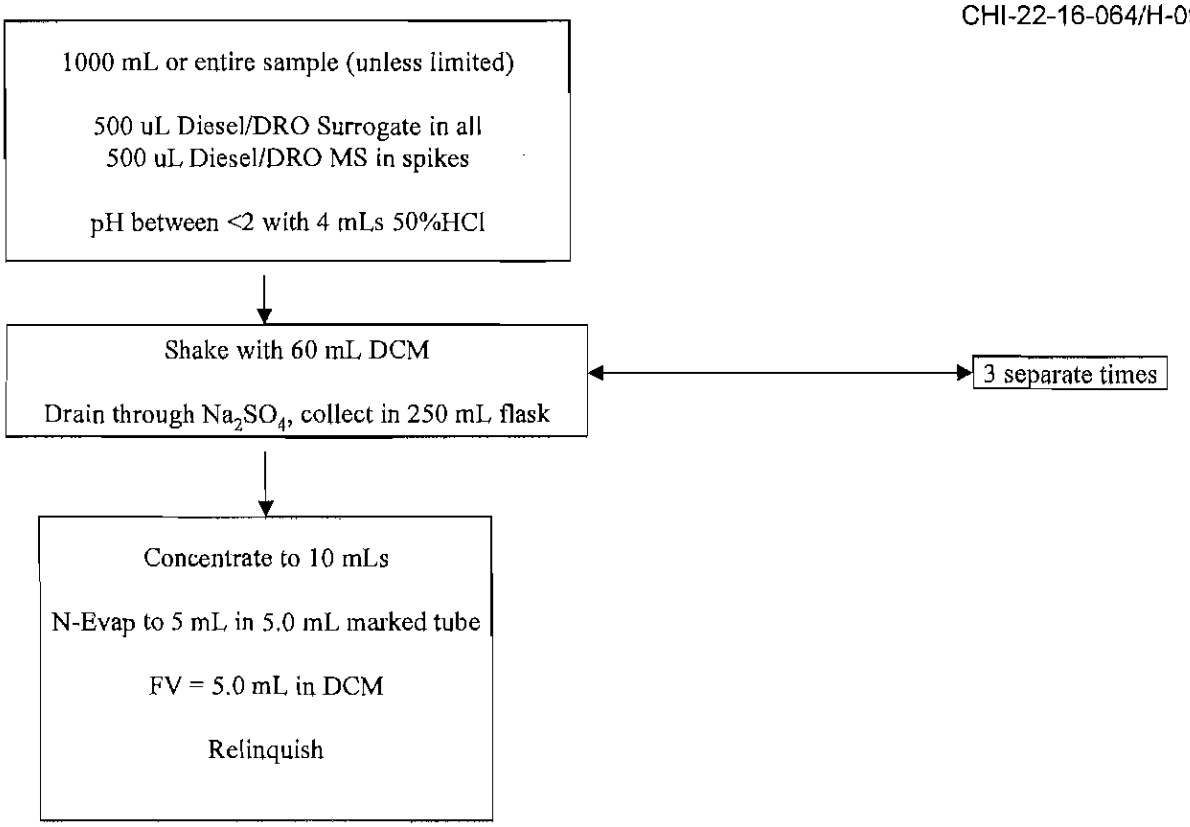
CHI-22-16-110\D-11/12



(028-010)

Flowchart for DRO Water (8015)
by 3510

CHI-22-16-064/H-09/12



(028-011)

Flowchart for DRO LVI Water (8015)
by 3510_LVI

CHI-22-16-126/B-02/13

250 mL or entire sample (unless limited)

50 uL Diesel/DRO Surrogate in all
50 uL Diesel/DRO MS in spikes

pH between <2 with 1 mLs 50%HCl

↓

Add 6g NaCl
Shake with 30 mL DCM
Drain through Na_2SO_4, collect in 250 mL flask

↔ 3 separate times

↓

Concentrate to 10 mLs

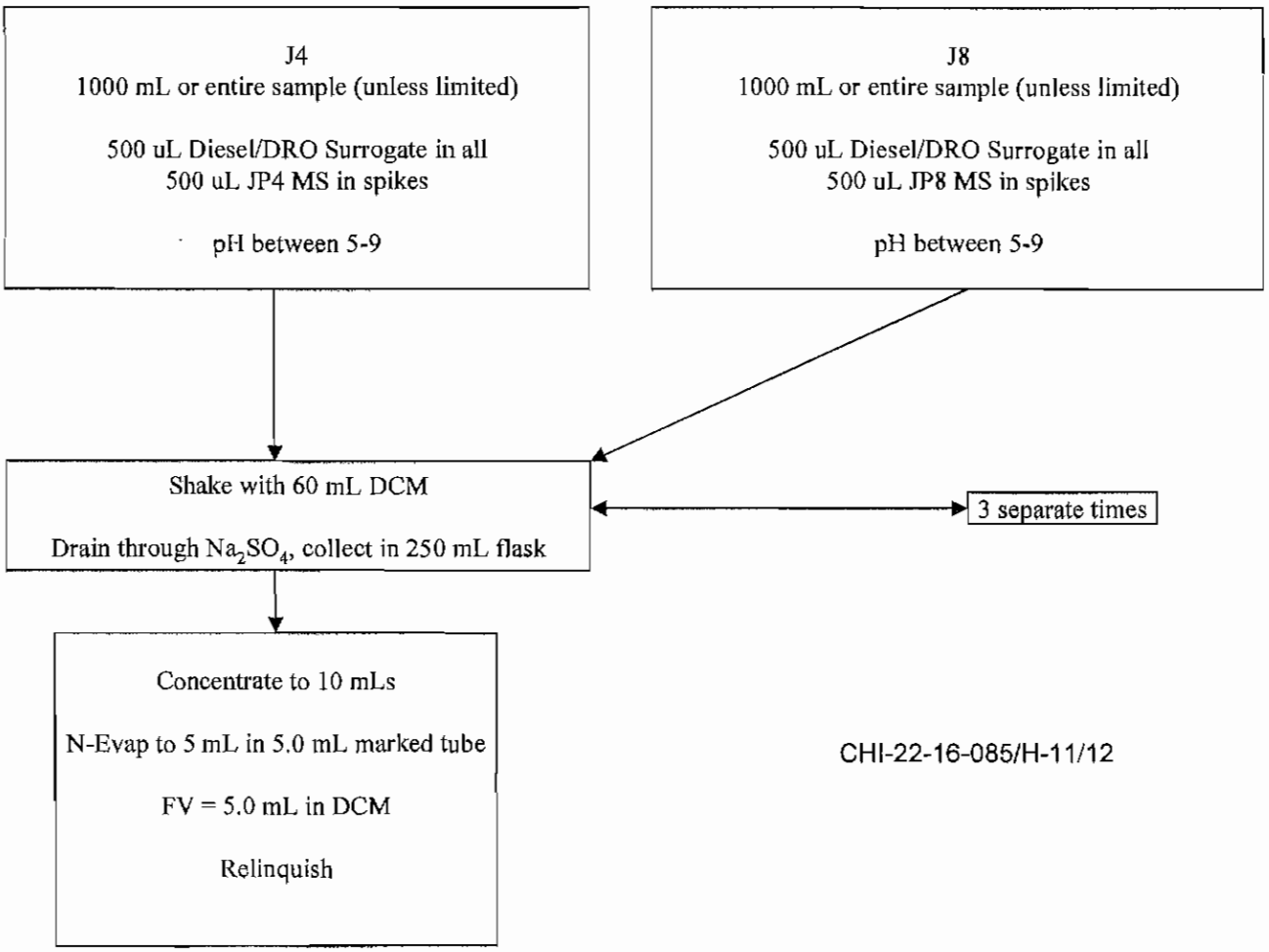
N-Evap to 5 mL in 2.5 mL marked tube

FV = 2.5 mL in DCM

Relinquish

(028-012)

Flowchart for JP4 Water
by 3510

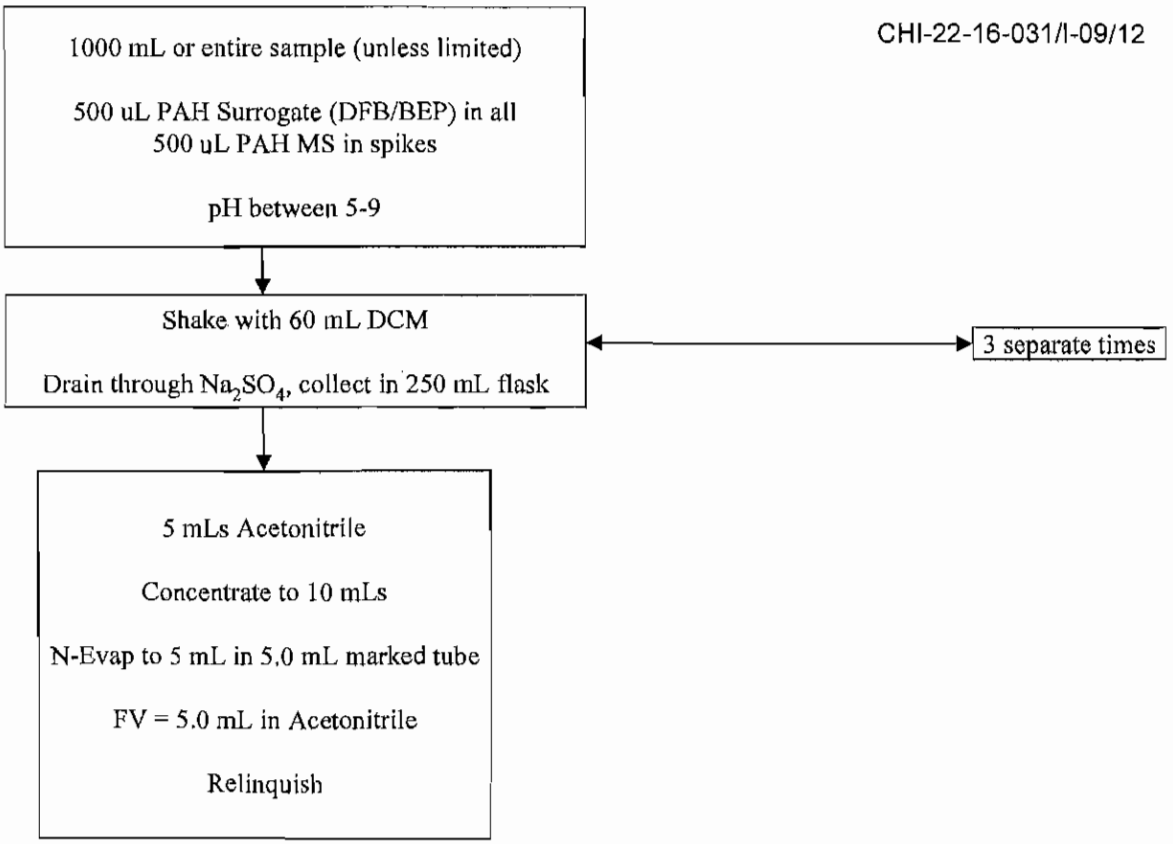


CHI-22-16-085/H-11/12

(028-013)

Flowchart for PAH Water (8310, 610)
by 3510

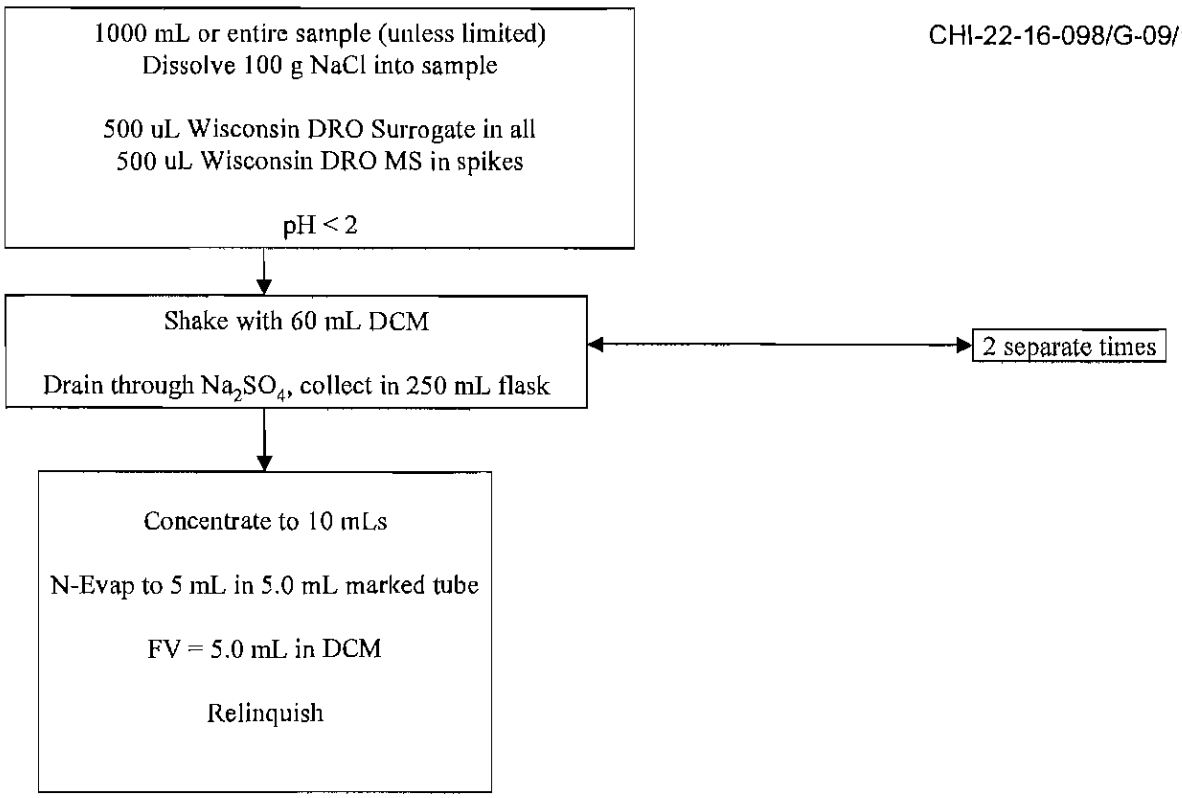
CHI-22-16-031/I-09/12



(028-014)

Flowchart for Wisconsin DRO Water
by 3510

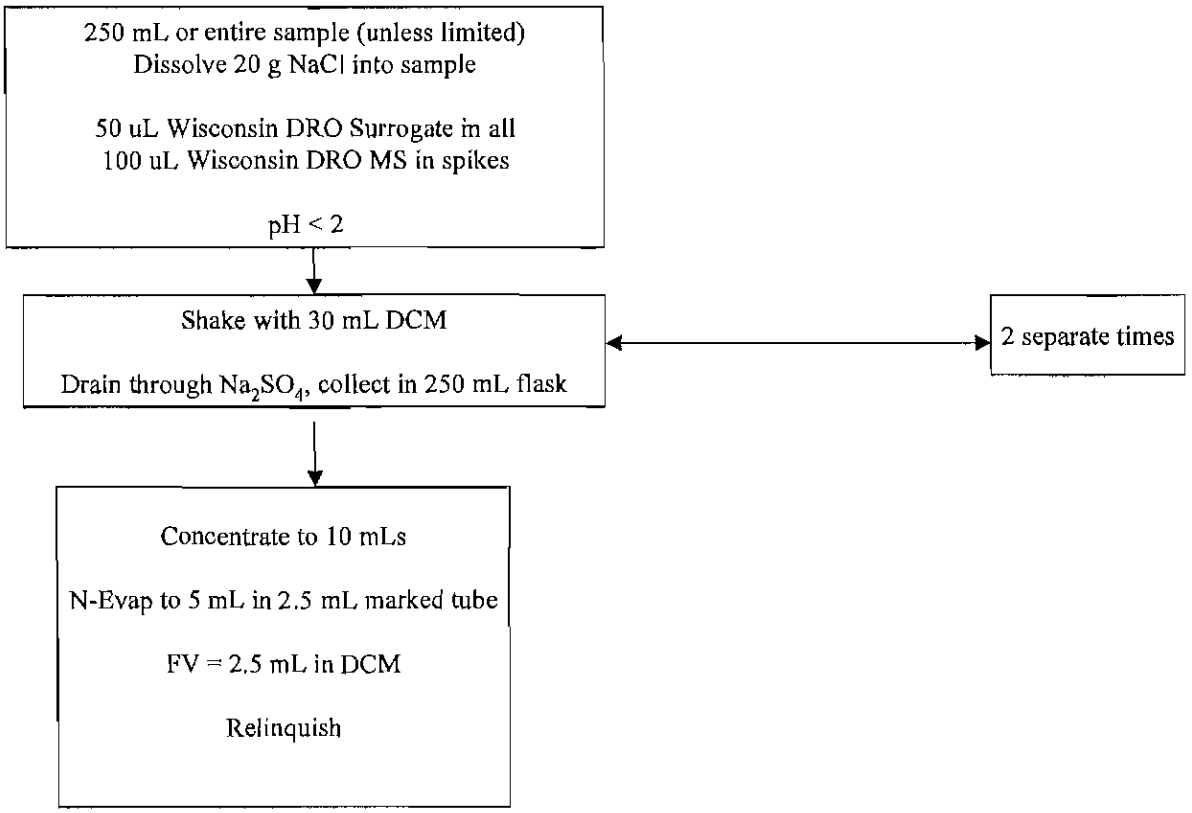
CHI-22-16-098/G-09/12



(028-015)

Flowchart for Wisconsin DRO LVI
Water
by 3510_LVI

CHI-22-16-128/C-2/13



(028-016)

Attachment 3:

Example: BNA Surrogate Stock Solution (029-001)

Certificate of Composition

DESCRIPTION: Semi-Volatile Acid/Base Surrogate Spike (High)

CATALOG NO.: 861142 MFG DATE: May-2012

LOT NO.: LB92759 EXPIRATION DATE: May-2015

SOLVENT: METHYLENE CHLORIDE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
NITROBENZENE-D5	4165-60-0	99.9	1000	LB47918
P-TERPHENYL-D14	1718-51-0	99.9	1000	LB83130
PHENOL-D6	13127-88-3	99.9	1500	LB85441
1,2-DICHLOROBENZENE-D4	2199-69-1	99.9	1000	LB83146
2-CHLOROPHENOL D4	93951-73-6	99.4	1500	LB74323
2-FLUOROBIPHENYL	321-60-8	99.9	1000	LB83498
2-FLUOROPHENOL	367-12-4	99.9	1500	LB83825
2,4,6-TRIBROMOPHENOL	118-79-6	99.9	1500	LB81262

2015987
ID: EXBNASUP_00029
Desc: BNA Surrogate Parent
Exp: 5/31/2015 Prpd/Rcvd: 11/26/2012
Sol: Prpd: knieriemend Op: 11/26/2012
BNA Surrogate Parent

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO Analytical
595 North Harrison Road • Bellefonte, PA 16823-0048 USA • Phone (814) 359-3441

(029-001)

Attachment 4:

Example: 8270 LCS Mix
(030-001 to 030-005)

Cerilliant
Analytical Reference Standards
A SIGMA-ALDRICH company

ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

Certificate of Analysis

8270 LCS Mix

Catalog Number:	ERS-077
Solution Lot:	ER111711-01
Expiration Date:	November 2016
Solvent:	Methanol::Methylene chloride::Benzene (70::25::5)
Volume per Ampule:	Not less than 5 mL
Storage:	Store in freezer.
Handling:	Sonicate for 5 minutes before use.
Intended Use:	For laboratory use only. Not suitable for human or animal consumption.
Safety:	Flammable, Carcinogen

2032985
ID: EXBNASPP_00085
Exp: 11/00/16 Prep: DAK
8270 LCS Mix Parent

Rec'd: 11/10/12

- Expiration Date has been established through real time stability studies.
- Ampules are overfilled to ensure a minimum 5 mL volume. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.

Traceability

- Gravimetrically prepared using qualified balances calibrated semi-annually by Mettler Toledo using NIST traceable weights. Calibration verification performed weekly and prior to each use utilizing NIST traceable weights. Each balance has been assigned a minimum weighing by Mettler Toledo taking into consideration the balance and installed environmental conditions to ensure weighing complies with USP tolerances of no more than 0.1% relative error.
- Concentration is verified against an independently prepared calibration solution gravimetrically prepared using balances calibrated to NIST.

Cerilliant certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration/retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.

ACLASS

Lara Sparks

Lara Sparks, Quality Assurance Director

May 16, 2012

Date

(030-001)

Component	Lot Number	CAS Number	Chemical Formula	Molecular Weight	Chromatographic Purity	Concentration
Acenaphthene	29697-41	83-32-9	$C_{12}H_{10}$	154.21	99.89%	200 µg/mL
Acenaphthylene	ER030707-01	208-96-8	$C_{12}H_8$	152.2	99.06%	200 µg/mL
Acetophenone	EB-1807	98-86-2	C_8H_8O	120.15	99.39%	200 µg/mL
Aniline	EB-1265	62-53-3	C_6H_7N	93.13	99.99%	200 µg/mL
Anthracene	ER061407-04	120-12-7	$C_{14}H_{10}$	178.23	99.24%	200 µg/mL
Azobenzene	P004554	103-33-3	$C_{12}H_{10}N_2$	182.22	98.97%	200 µg/mL
Benz(a)anthracene	ER121707-01	56-55-3	$C_{18}H_{12}$	228.29	99.12%	200 µg/mL
Benzidine [1]	PR081108-03	92-87-5	$C_{12}H_{12}N_1$	184.24	99.96%	200 µg/mL
Benzo(b)fluoranthene	ER022008-02	205-99-2	$C_{20}H_{12}$	252.32	99.95%	200 µg/mL
Benzo(k)fluoranthene	ER061608-02	207-08-9	$C_{20}H_{12}$	252.32	99.96%	200 µg/mL
Benzo(g,h,i)perylene	ER020708-08	191-24-2	$C_{22}H_{12}$	276.34	99.29%	200 µg/mL
Benzo(a)pyrene	ER071309-02	50-32-8	$C_{20}H_{12}$	252.32	99.55%	200 µg/mL
Benzoic acid	PR090308-01	65-85-0	$C_7H_6O_2$	122.12	99.99%	200 µg/mL
Benzyl alcohol	EB-1963	100-51-6	C_7H_8O	108.14	99.74%	200 µg/mL
Biphenyl	PER_053_97109	92-52-4	$C_{12}H_{10}$	154.21	99.70%	200 µg/mL
Bis(2-chloroethoxy)methane	P004590	111-91-1	$C_5H_{10}O_2Cl_2$	173.04	97.60%	200 µg/mL
Bis(2-chloroethyl)ether	PER_456_102708	111-44-4	$C_4H_8OCl_2$	143.01	99.79%	200 µg/mL
Bis(1-chloroisopropyl)ether	PR073110-01	108-60-1	$C_6H_{12}OCl_2$	171.07	99.37%	200 µg/mL
Bis(2-ethylhexyl) phthalate	P004582	117-81-7	$C_{24}H_{38}O_4$	390.56	99.17%	200 µg/mL
4-Bromophenyl phenyl ether	P004550	101-55-3	$C_{12}H_9OBr$	249.11	98.88%	200 µg/mL
Butyl benzyl phthalate	EB-2108	85-68-7	$C_{19}H_{20}O_4$	312.37	98.03%	200 µg/mL
Carbazole	P004638	86-74-8	$C_{12}H_9N$	167.21	95.37%	200 µg/mL
p-Chloroaniline	EB-1801	106-47-8	C_6H_6NCl	127.57	99.91%	200 µg/mL
4-Chloro-3-methylphenol	PR060807-01	59-50-7	C_7H_7OCl	142.58	99.96%	200 µg/mL
2-Chloronaphthalene	P004640	91-58-7	$C_{10}H_7Cl$	162.62	99.21%	200 µg/mL
2-Chlorophenol	PR060807-02	95-57-8	C_6H_4OCl	128.56	99.43%	200 µg/mL
4-Chlorophenyl phenyl ether	P004559	7005-72-3	$C_{12}H_9OCl$	204.66	98.13%	200 µg/mL
Chrysene	ER120810-02	218-01-9	$C_{18}H_{12}$	228.29	98.66%	200 µg/mL
Dibenz(a,h)anthracene	ER102009-03	53-70-3	$C_{22}H_{14}$	278.35	99.83%	200 µg/mL
Dibenzofuran	P004552	132-64-9	$C_{12}H_8O$	168.19	99.49%	200 µg/mL
Di-n-butyl Phthalate	P004541	84-74-2	$C_{16}H_{22}O_4$	278.35	99.72%	200 µg/mL
1,2-Dichlorobenzene	EB-2097	95-50-1	$C_6H_4Cl_2$	147	99.47%	200 µg/mL
1,3-Dichlorobenzene	EB-1908	541-73-1	$C_6H_4Cl_2$	147	99.63%	200 µg/mL
1,4-Dichlorobenzene	PER_226_104287	106-46-7	$C_6H_4Cl_2$	147	99.98%	200 µg/mL
3,3'-Dichlorobenzidine	ER083107-03	91-94-1	$C_{12}H_{10}Cl_2N_2$	253.13	99.67%	200 µg/mL
2,4-Dichlorophenol	P004712	120-83-2C	$_6H_4OCl_2$	163	99.07%	200 µg/mL
2,6-Dichlorophenol	P004533	87-65-0	$C_6H_4OCl_2$	163	99.99%	200 µg/mL
Diethyl Phthalate	PER_096_102706	84-66-2	$C_{12}H_{14}O_4$	222.24	99.96%	200 µg/mL
2,4-Dimethylphenol	P004557	105-667-9	$C_8H_{10}O$	122.17	99.62%	200 µg/mL
Dimethyl Phthalate	EB-1774	131-11-3	$C_{10}H_{10}O_4$	194.19	99.94%	200 µg/mL
2,4-Dinitrophenol	PR062607-03	51-28-5	$C_6H_4N_2O_5$	184.11	99.84%	200 µg/mL
2,4-Dinitrotoluene	EB-1379	121-14-2	$C_7H_6N_2O_4$	182.14	99.00%	200 µg/mL
2,6-Dinitrotoluene	PER_083_93401	606-20-2	$C_7H_6N_2O_4$	182.14	99.59%	200 µg/mL
Dinoseb	ER110209-01	88-85-7	$C_{10}H_{12}N_2O_5$	240.22	99.96%	200 µg/mL
Di-n-octyl Phthalate	PR092710-01	117-84-0	$C_{24}H_{38}O_4$	390.56	99.37%	200 µg/mL

Component	Lot Number	CAS Number	Chemical Formula	Molecular Weight	Chromatographic Purity	Concentration
Diphenyl ether	P004715	101-84-8	$C_{12}H_{10}O$	170.21	99.98%	200 µg/mL
1,4-Dioxane	PR052809-05	123-91-1	$C_4H_8O_2$	88.11	99.95%	200 µg/mL
2-Ethoxyethanol	PER_052_102546	110-80-5	$C_4H_{10}O_2$	90.12	99.96%	200 µg/mL
Fluoranthene	PN111910-02	206-44-0	$C_{16}H_{10}$	202.26	98.45%	200 µg/mL
Fluorene	35001-29	86-73-7	$C_{13}H_{10}$	166.22	98.95%	200 µg/mL
Hexachlorobenzene	P004546	118-74-1	C_6Cl_6	284.78	99.71%	200 µg/mL
Hexachloro-(1,3)-butadiene	P141819	87-68-3	C_4Cl_6	260.76	97.28%	200 µg/mL
Hexachlorocyclopentadiene	PR081210-01	77-47-4	C_5Cl_6	272.77	96.84%	200 µg/mL
Hexachloroethane	EB-2003	67-72-1	C_2Cl_6	236.74	99.57%	200 µg/mL
Hexachlorophene [1]	P004584	70-30-4	$C_{13}H_6O_2Cl_6$	406.91	94.70%	200 µg/mL
Hexachloropropene	EB-1977	1888-71-7	C_3Cl_6	248.75	96.66%	200 µg/mL
Indeno(1,2,3,-c,d)pyrene	ER082107-02	193-39-5	$C_{22}H_{12}$	276.34	99.42%	200 µg/mL
Isophorone	EB-1778	78-59-1	$C_9H_{14}O$	138.21	98.21%	200 µg/mL
2-Methyl-4,6-dinitrophenol	P004549	534-52-1	$C_7H_6N_2O_5$	198.14	99.77%	200 µg/mL
2-Methylnaphthalene	EB-1779	91-57-6	$C_{11}H_{10}$	142.20	97.52%	200 µg/mL
2-Methylphenol	PR061107-01	95-48-7	C_7H_8O	108.14	99.55%	200 µg/mL
4-Methylphenol	PER_016_99798	106-44-5	C_7H_8O	108.14	99.68%	200 µg/mL
Naphthalene	PR050410-01	91-20-3	$C_{10}H_8$	128.17	99.88%	200 µg/mL
2-Nitroaniline	P004682	88-74-4	$C_6H_6N_2O_2$	138.13	99.66%	200 µg/mL
3-Nitroaniline	P004543	99-09-2	$C_6H_6N_2O_2$	138.13	99.90%	200 µg/mL
4-Nitroaniline	EB-1796	100-01-6	$C_6H_6N_2O_2$	138.13	99.77%	200 µg/mL
Nitrobenzene	PR090310-01	98-95-3	$C_6H_5NO_2$	123.11	99.96%	200 µg/mL
2-Nitrophenol	PR060807-03	88-75-5	$C_6H_5NO_3$	139.11	99.84%	200 µg/mL
4-Nitrophenol	P004713	100-02-7	$C_6H_5NO_3$	139.11	99.93%	200 µg/mL
N-Nitrosodi-n-butylamine	P004561	924-16-3	$C_8H_{18}N_2O$	158.24	99.78%	200 µg/mL
N-Nitrosodiethylamine	P004558	55-18-5	$C_4H_{10}N_2O$	102.14	99.80%	200 µg/mL
N-Nitrosodimethylamine	P004569	62-75-9	$C_2H_6N_2O$	74.08	100.00%	200 µg/mL
N-Nitrosodi-n-propylamine	P004632	621-64-7	$C_6H_{14}N_2O$	130.19	99.95%	200 µg/mL
N-Nitrosodiphenylamine	PR102606-01	86-30-6	$C_{12}H_{10}N_2O$	198.22	96.79%	200 µg/mL
N-Nitrosomethylethylamine	PR073110-02	10595-95-6	$C_3H_8N_2O$	88.11	99.41%	200 µg/mL
N-Nitrosopiperidine	P004545	100-75-4	$C_5H_{10}N_2O$	114.15	99.45%	200 µg/mL
N-Nitrosopyrrolidine	P004551	930-55-2	$C_4H_8N_2O$	100.12	99.94%	200 µg/mL
Pentachlorobenzene	P004714	608-93-5	C_6HCl_5	250.34	99.56%	200 µg/mL
Pentachloroethane	EB-1954	76-01-7	C_2HCl_5	202.29	98.30%	200 µg/mL
Pentachlorophenol	P004716	87-86-5	C_6HCl_5O	266.34	98.76%	200 µg/mL
Phenanthrene	ER042805-02	85-01-8	$C_{14}H_{10}$	178.23	99.19%	200 µg/mL
Phenol	PR092309-03	108-95-2	C_6H_6O	94.11	99.97%	200 µg/mL
Pyrene	ER050305-03	129-00-0	$C_{16}H_{10}$	202.26	99.47%	200 µg/mL
Pyridine	PER_039_102530	110-86-1	C_5H_5N	79.10	99.95%	200 µg/mL
1,2,4,5-Tetrachlorobenzene	EB-1955	95-94-3	$C_6H_2Cl_4$	215.89	99.34%	200 µg/mL
2,3,4,6-Tetrachlorophenol	ER100206-01	58-90-2	$C_6H_2Cl_4O$	231.89	99.28%	200 µg/mL
1,2,4-Trichlorobenzene	EB-1677	120-82-1	$C_6H_3Cl_3$	181.45	99.73%	200 µg/mL
2,4,5-Trichlorophenol	PR040307-02	95-95-4	$C_6H_3Cl_3O$	197.45	98.20%	200 µg/mL
2,4,6-Trichlorophenol	P004555	88-06-2	$C_6H_3Cl_3O$	197.45	98.95%	200 µg/mL

[1] Due to stability problems, it is recommended not to use this analyte for quantitation.

(030-003)

Standard Solution Assay Parameters

Analysis Method:	GC/MS
Column:	DB-5ms, 30 m x 0.25 mm ID, 0.25 µm film thickness
Temp Program:	40°C (hold 2 mins) to 300°C at 10°C/min (hold 7 mins)
Injector Temp:	Cool on-column
Detector Temp:	280°C

Calibration Curve

Calibration Curve:	Linear Regression
Number of Points:	1

Solution Standard Verification and Homogeneity

Compound	Verified Concentration µg/mL	Acceptance Criteria	%RSD - Homogeneity %	Acceptance Criteria
Acenaphthene	190.7	± 10%	0.7	≤ 5%
Benzo(a)pyrene	190.4	± 10%	1.3	≤ 5%
Benzyl alcohol	190.0	± 10%	3.1	≤ 5%
Bis(2-ethylhexyl)phthalate	189.0	± 10%	3.6	≤ 5%
Carbazole	189.4	± 10%	3.9	≤ 5%
4-Chloroaniline	187.7	± 10%	4.1	≤ 5%
4-Chloro-3-methylphenol	189.5	± 10%	2.7	≤ 5%
Dibenzofuran	189.0	± 10%	1.1	≤ 5%
1,4-Dichlorobenzene	189.5	± 10%	1.2	≤ 5%
3,3'-Dichlorobenzidine	192.6	± 10%	2.4	≤ 5%
2,4-Dichlorophenol	189.3	± 10%	2.2	≤ 5%
2,4-Dinitrotoluene	189.4	± 10%	4.1	≤ 5%
Di-n-octyl-phthalate	191.7	± 10%	1.1	≤ 5%
Fluoranthene	190.8	± 10%	2.2	≤ 5%
Hexachloro-(1,3)-butadiene	188.7	± 10%	1.0	≤ 5%
Isophorone	191.5	± 10%	3.1	≤ 5%
2-Methylnaphthalene	189.8	± 10%	1.0	≤ 5%
3-Nitroaniline	188.1	± 10%	1.8	≤ 5%
2-Nitrophenol	186.8	± 10%	3.6	≤ 5%
Nitrobenzene	192.3	± 10%	1.6	≤ 5%
N-Nitrosodi-n-butylamine	189.8	± 10%	2.7	≤ 5%
N-Nitrosodiphenylamine	189.8	± 10%	2.4	≤ 5%
Pentachlorobenzene	188.1	± 10%	1.4	≤ 5%
Phenol	191.1	± 10%	1.4	≤ 5%
Pyridine	189.8	± 10%	2.6	≤ 5%

- Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration solution.
- Homogeneity is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. The %RSD of samples pulled from across the lot demonstrate homogeneity.
- This standard was prepared from six stocks that have been verified for accuracy and homogeneity. Verification of this standard was performed by monitoring 25 components listed in the table above, including Method 8270 Check Compounds (CCC) and at least one analyte from each stock.

(030-004)

COA Revision History

Revision No.	Date	Reason for Revision
00	12/21/2011	Initial version
01	4/11/2012	Missing footnote was added.
		Added statement that solution should be sonicated prior to use.
02	5/16/2012	Corrected "Bis(2-chloroisopropyl)ether" (CAS 108-60-1) to "Bis(1-chloroisopropyl)ether"

(030-005)

Attachment 5:

Example: 3,3'-Dimethylbenzidine (031-001 to 031-004)

Cerilliant®
Analytical Reference Standards
a SIGMA-ALDRICH company

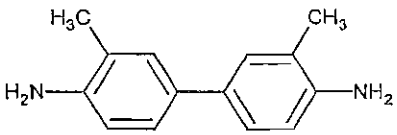
ISO GUIDE 34
ISO/IEC 17025
ISO 13485
ISO 9001
GMP/GLP

Certificate of Analysis

3,3'-Dimethylbenzidine

2033995
10' EXDMBSPP_00077
Exp: 06/31/17 Prep: DAK
BNA LCS 3,3'-Dimethylbenz

Rec'd: 12/10/12



Catalog Number:	ERD-132S
Solution Lot:	ER013012-08
Expiration Date:	May 2017
Solvent:	Methylene chloride
Volume per Ampule:	Not less than 1.2 mL
Storage:	Store unopened in freezer.
Shipping:	Ambient. See Stability Section.
Intended Use:	For R&D/ analytical purposes only. Not suitable for human or animal consumption.
Safety:	Carcinogen

- Expiration Date has been established through real time stability studies.
- Ampules are overfilled to ensure a minimum 1.2 mL volume fill. We advise laboratories to use measured volumes of this standard solution before diluting to the desired concentration.

Component	Solution Purity	Certified Concentration
3,3'-Dimethylbenzidine	99.6%	2000 ± 13 µg/mL

- Uncertainty of the concentration is expressed as an expanded uncertainty in accordance with ISO 17025 and Guide 34 at the approximate 95% confidence interval using a coverage factor of k = 2 and has been calculated by statistical analysis of our production system and incorporates uncertainty of the purity factor, material density, and balance and weighing technique.
- This standard is prepared gravimetrically and mass results are reported on the conventional basis for weighing in air. Concentration is calculated based on: the actual measured mass; Purity Factor of the analyte(s); measured mass of the solution; and the density of the pure diluent at 20°C.
- Concentration is corrected for chromatographic purity, residual water, residual solvents and residual inorganics.

Solution Standard Verification and Homogeneity

Standard Solution	Lot Number	Verified Concentration (µg/mL) Actual Results	Acceptance Crtieria	%RSD - Homogeneity Actual Results	Acceptance Crtieria
New Lot	ER013012-08	1999	± 3%	1.0	≤ 3%
Previous Lot	ER061710-01	2017	± 3%	2.5	≤ 3%

- Concentration is verified through multiple analyses and is calculated as the average of multiple analyses compared to an independently prepared calibration solution.
- Homogeneity of the New Lot is ensured through rigorous production process controls statistically analyzed to evaluate risk and verified by analysis. The % RSD of samples pulled from across the lot demonstrate homogeneity of the New Lot.
- The % RSD of the Previous Lot represents variability of the analysis performed at the time of release.

Traceability

- Gravimetrically prepared using qualified balances calibrated semi-annually by Mettler Toledo using NIST traceable weights. Calibration verification performed weekly and prior to each use utilizing NIST traceable weights. Each balance has been assigned a minimum weighing by Mettler Toledo taking into consideration the balance and installed environmental conditions to ensure weighing complies with USP tolerances of no more than 0.1% relative error.
- Concentration is verified against an independently prepared calibration solution gravimetrically prepared using balances calibrated to NIST.
- In addition, each neat material utilized has been identified and thoroughly characterized through the use of multiple analytical techniques. Spectral data is provided on subsequent pages of the COA.

Cerilliant certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration/retest date when stored unopened as recommended. Product should be used shortly after opening to avoid concentration changes due to evaporation. Warranty does not apply to ampoules stored after opening.

ACCLASS
REFERENCE MATERIAL PRODUCER

Lara Sparks

Lara Sparks, Quality Assurance Director

June 12, 2012
Date

(031-001)

Standard Solution Assay Parameters

Analysis Method:	GC/FID
Column:	DB-5ms, 30 m x 0.53 mm ID, 1.5 µm film thickness
Temp Program:	60°C to 280°C at 40°C/min, 7.0 min hold
Injector Temp:	Cool-on-column
Detector Temp:	325°C

Calibration Curve

Calibration Curve:	Linear Regression
Number of Points:	4
Linearity (r):	0.999

Neat Material Data

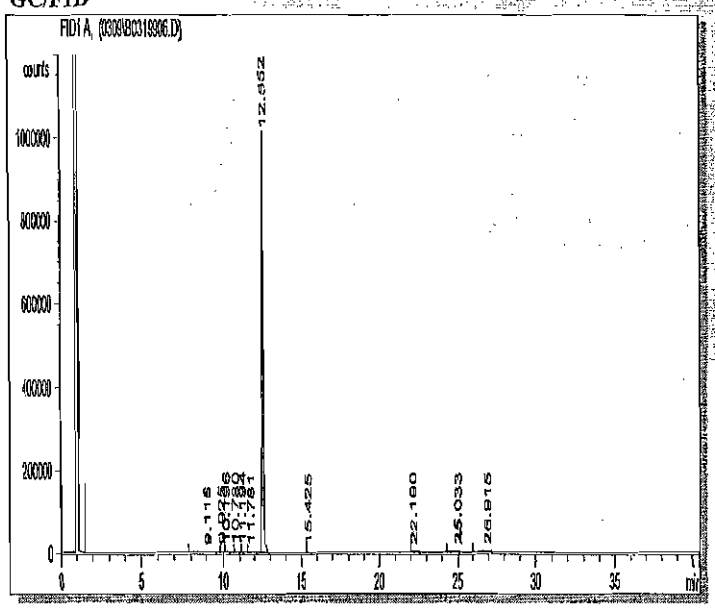
Compound Name:	3,3'-Dimethylbenzidine	**Chemical Formula:**	$C_{14}H_{16}N_2$
Compound Lot:	PR031309-01	**CAS Number:**	119-93-7
		Molecular Weight:	212.29

Neat Material Characterization Summary

Analytical Test	Method	Results
Chromatographic Purity by GC/FID Analysis	SP10-0101	99.5%
Identity by GC/MS Analysis	SP10-0105	Consistent with Structure
Purity Factor		99.55%

- Chromatographic purity is calculated as the average of two independently performed analyses utilizing two different methods. Acceptance criteria requires the purity values to be within 0.5% of each other.
- The chromatographic purity value is used to calculate the Purity Factor.

GC/FID



Column:	DB-5ms, 30 m x 0.53 mm ID, 1.5 µm film thickness
Temp Program:	40°C to 200°C at 40°C/min 200°C to 245°C at 5°C/min 245°C to 280°C at 2°C/min (hold 10 min)
Injector Temp:	Cool-on-Column
Detector Temp:	325°C
Data File Name:	S:\GC\GC2\2009\0309\B0319906.D
Operator:	RPC
Instrument:	GC#2
Sample Name:	PR031309-01
Method File:	PER064.M
Acquired:	March 19, 2009 12:05 PM

Peak #	Ret Time	Area	Height	Area %
1	9.12	1202	63	0.02
2	9.93	835	203	0.02
3	10.20	9187	2059	0.17
4	10.78	5838	1230	0.10
5	11.19	849	206	0.02
6	11.78	1076	80	0.02
7	12.55	5538950	1011080	99.53
8	15.43	142	19	0.00
9	22.19	1487	75	0.03
10	25.03	1662	46	0.03
11	26.92	3608	135	0.06

(031-002)

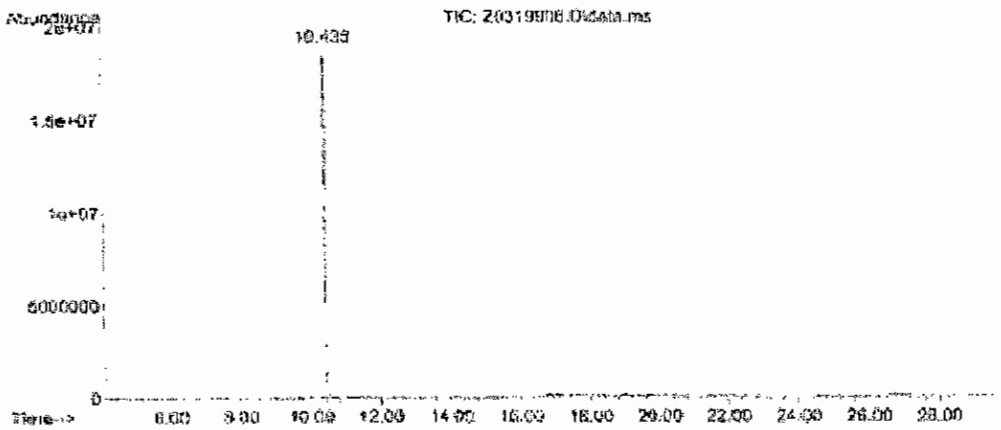
Spectral and Physical Data (cont.)

Identity by GC/MS Analysis

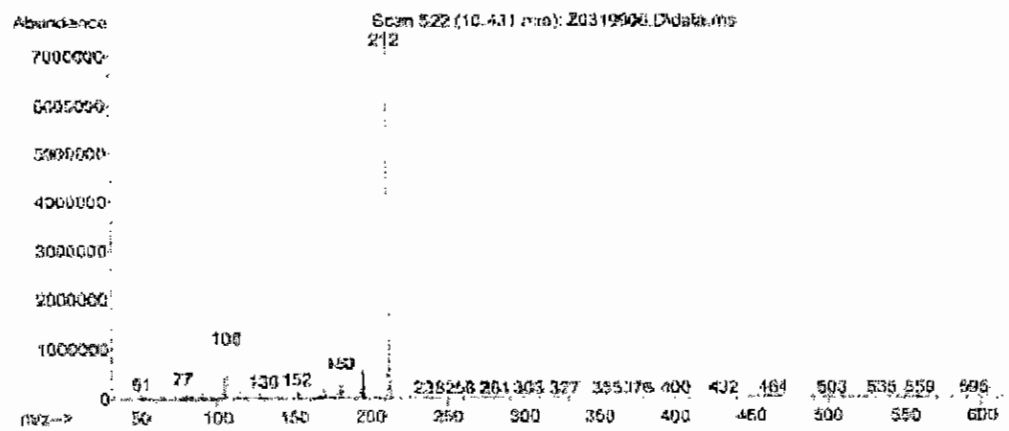
Compound Name : 3,3'-Dimethylbenzidine
Lot Number : PR031309-01
Instrument : Agilent 5975B MSD/6890N GC
Operator-Inst ID : RPC - C10106
Date Reported : Fri Mar 20 09:00:35 2009
Column Type : DB-5ms, 20m x 0.25mm ID, 0.25um film thickness
Temp. Program : 50°C to 200°C@40°C/min, 200°C to 300°C@10°C/min, 1.6min hold
Injector Temp. : Cool on-column
Carrier Gas : Helium
Flow Rate (mL/min) : 0.80 mL/min
Transfer Line Temp. : 280°C

Scan Range : 50-600

Total Ion Chromatogram



Mass Spectrum



(031-003)

Stability

Short Term Stability : A summary of accelerated stability findings for this product is listed below.		
Storage Condition	**Mean Kinetic Temperature (MKT)**	**Time Period**
Freezer	-15°C	No decrease in purity was noted after one week.
Refrigerator	4°C	
Room Temperature	21°C	
40°C	40°C	
Transport/Shipping : *Stability data supports transport of this product at ambient conditions.*		
Short Term Storage: *Stability data supports short term storage up to 1 year at Refrigerate conditions.*		

Appendix 1

BNA Halowax

Other than having different standards, the BNA Halowax extraction is identical to the BNA separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 BNA Surrogate Spike (SIM) Solution

The surrogate used for the halowax extraction is the same solution used for the BNA SIM extraction. The BNA Surrogate Spike (SIM) Solution is prepared from the BNA Surrogate Spike at the levels listed below. The BNA Surrogate (SIM) Spike Solution is added to all samples, MBs, LCS and MS/MSDs.

Compound	Concentration (ug/mL)
p-Terphenyl-d_4	10
Nitrobenzene-d_5	10
2-Fluorobiphenyl	10
1,2-Dichlorobenzene-d_4	10
Phenol-d_5	15
2-Fluorophenol	15
2,4,6-Tribromophenol	15
2-Chlorophenol-d_4	15

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

1.2 BNA Halowax Matrix Spike Solution

The BNA Halowax Matrix Spike Solution is prepared by the BNA analytical group. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. The matrix spike solution is added to the LCS and MS/MSD.

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

Appendix 2

Alachlor/Atrazine

Other than having different standards, the Alachlor/Atrazine extraction is identical to the pesticide separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 Pesticide/PCB Surrogate Spike Solution

The surrogate used for the Alachlor and Atrazine extraction is the same solution used for the pesticides/PCBs extraction. The surrogate used in the extraction of pesticides/PCBs from waters is a solution of Decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachloro-m-xylene (TCX) in acetone at the concentrations listed below. The Pesticide/PCB Surrogate Spike Solution is added to all samples, MBs, LCS and MS/MSD.

Compound	Concentration (ug/mL)
DCB	0.4
TCX	0.4

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 Alachlor/Atrazine Matrix Spike Solution

The LCS/MS solution used in the extraction Alachlor and Atrazine from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. The Alachlor/Atrazine Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

Appendix 3

Wisconsin DRO

In addition using specific surrogate and spike solutions, the Wisconsin DRO extraction differs from the SW-846 DRO extraction. The changes are noted in this appendix.

1.0 Reagents

1.1 Heat-Purified Sodium Chloride

Sodium chloride purchased directly from vendor. Purify by heating at 400°C for four hours in a shallow drying tray, cool in a desiccator, and store in a glass bottle.

- Life of Reagent: 1-year.
- Storage Requirements: Store in a glass container and keep dry (anhydrous).

2.0 Surrogate and Matrix Spike Solutions

2.1 Wisconsin DRO Surrogate Solution

The Wisconsin DRO surrogate solution is prepared by the DRO analyst at the levels listed below. The surrogate is added to all samples, MBs, LCS/LCD and MS/MSDs.

Compound	Concentration (ug/mL)
C9-Nonane	100

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

2.2 Wisconsin DRO Matrix Spike Solution

The Wisconsin DRO Matrix Spike Solution is prepared by the DRO analyst. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. The matrix spike solution is added to the LCS/LCD and MS/MSD.

- Life of Standard: 6-months from the preparation date (as documented from the vendor).
- Storage Requirements: As stated in Section 5.2

3.0 Wisconsin DRO Extraction

3.1 Clarification of section MB and LCS water preparation in **7.6.1**. Add 1.0 L (250 mLs for LVI) of Milli-Q water to 3 separatory funnels and label as the MB, LCS and LCD. A LCD must be extracted even if there is a MS/MSD.

3.2 Add to section **7.6.3**. Samples should be preserved in the field. If sample pH is greater than two, sample results must be flagged. Add 4 mLs of the 50% hydrochloric acid to the MB, LCS, and LCSD.

3.3 Add to section **7.6.4**. Add 100 g (20 g for LVI) NaCl to separtory funnel, then shake. Ensure that the salt is dissolved into sample before adding surrogate and spike.

3.4 In section **7.6.11**, repeat the extraction only once more for a total of two, 60 mL (30 mL for LVI) extractions.

3.5 Make the following changes in section **7.6.12.1**. Use the 10 mL insulated concentrator tubes. The K-D bath should be at 70° C.

3.6 In section **7.6.12**, add rinse a small amount of DCM through the Snyder column before removing it from the K-D flask.

3.7 Refer to Diesel/DRO section in **7.6.14**.

Appendix 4

JP4 and JP8

Other than having different standards, the JP4 or JP8 extraction is identical to the DRO separatory funnel procedure.

1.0 Surrogate and Matrix Spike Solutions

1.1 DRO Surrogate Spike Solution

The surrogate used for the JP4 or JP8 extraction is the same solution used for the DRO extraction. The surrogate used in the extraction of DRO from waters is a solution of 2-Fluorobiphenyl and o-Terphenyl in acetone at the concentrations listed below. The GC group prepares the working surrogate solution. The DRO Surrogate Spike Solution is added to all samples, MBs, LCS and MS/MSD.

Compound	Concentration (ug/mL)
2-Fluorobiphenyl	100
o-Terphenyl	100

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 JP4 Matrix Spike Solution

The LCS/MS solution used in the extraction of JP4 from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. The JP4 Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.2 JP8 Matrix Spike Solution

The LCS/MS solution used in the extraction of JP8 from waters is a solution in methanol. The composition and concentration of this standard is beyond the scope of this SOP. See the analytical SOP for specifics about this standard. The JP8 Spike Solution is added to LCS and MS/MSD.

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

Appendix 5

AR1242 and AR1254 PCBs

Other than having different matrix spike standards, the AR1242 and AR1254 extraction is identical to the PCB water extraction.

1.0 Matrix Spike Solutions

These two matrix spike solutions need to be used separately. Samples batches will contain an LCS/MS/MSD for AR1242 and an LCS/MS/MSD for AR1254.

1.1 AR1242 Spike Solution

The LCS/MS solution used in the extraction of PCBs from soil/sediments is a solution of Arochlor 1242 in methanol at the following concentrations. The PCB Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
AR 1242	5.0

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

1.1 AR1254 Spike Solution

The LCS/MS solution used in the extraction of PCBs from soil/sediments is a solution of Arochlor 1254 in methanol at the following concentrations. The PCB Spike Solution is added to the LCS and MS/MSD.

Compound	Concentration (ug/mL)
AR 1242	5.0

- Life of Standard: Same as parent solution.
- Storage Requirements: As stated in Section 5.2.

TITLE: Wet Chemistry Flash Point

Approvals (Signature/Date):

Carla Bonner 11/8/12
Carla Bonner — Date
Supervisor, Wet Chemistry

Diane L. Harper 11/8/12
Diane L. Harper — Date
Inorganics Manager

Terese A. Preston 11/8/12
Terese A. Preston — Date
Quality Assurance Manager

Mark Storm 11/8/12
Mark Storm — Date
Env. Health & Safety Coor.

Michael J. Healy 11/8/12
Michael J. Healy — Date
Laboratory Director

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1.0 SCOPE / APPLICATION

This Standard Operating Procedure (SOP) outlines the procedure for determining the flash point of samples using closed-cup and open-cup techniques. It was written using SW-846, 3rd Edition (1986), Method 1010, Update 4 Method 1010A, and ASTM D-93 as references for the closed-cup procedure and ASTM D-92 for open-cup flash point.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e. PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP) and authorized via laboratory signature approval; and amended to the data packages case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Since temperature is an open-ended scale, there is no conventional detection limit for flash points.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Flash point readings are limited by the initial temperature to which the sample may be taken. The TestAmerica Chicago closed-cup tester is programmed to start applying the ignitor 41° F below the specified expected flash point and continue to 36° F above that temperature, or until a flash is detected, whichever is lower. A non-flash sample will be reported as >176° F for routine closed-cup samples. The open-cup tester is manual and the analyst generally starts applying the flame to a cooled sample aliquot and continues to 200 °F or until the sample boils or flashes, whichever occurs first.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA_QAM).

1.2 Summary of Method

Methods 1010 and 1010A use the Pensky-Martens closed-cup flash point tester technique to determine the flash point of liquids including those that tend to form a surface film under test conditions, according to ASTM D-93, method A or B. Liquids containing non-filterable, suspended solids shall also be tested using this method. Crushed solid materials or soils can be analyzed in the closed cup instrument by method B with the stirrer removed, but this is a modification of the method that must be documented.

The sample is heated in an automated closed-cup system at a slow, constant rate with continual stirring (where applicable, i.e., soils and solid samples are not stirred). An electrical igniter coil is automatically dipped into the cup at regular intervals with simultaneous interruption of stirring. The lowest temperature at which application of the coil causes the vapors above the test sample to ignite is recorded as the flash point. The instrument detects the flash point by the sudden increase in temperature. Flash point results are automatically corrected for the barometric pressure by a sensor and software in the instrument.

The open cup is filled to the specified mark with the sample. The temperature is increased rapidly at first, then slower at a constant rate. A small flame is passed across the cup at regular intervals. The flash point is recorded as the lowest temperature that ignites the vapors above the surface of the sample.

2.0 INTERFERENCES

- Drafts, incorrect starting temperature, incorrect heating rate, and incorrect stirring rates will all affect the observed flash point. The closed cup instrument is programmed to operate within the specifications of the method chosen (A or B) on the keyboard at the beginning of the test.
- It should be noted that some samples may contain only a trace of ignitable solvent and will only flash once or a few times even if the temperature is raised.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- In the event a sample ignites and continues burning in the test apparatus do not attempt to remove the sample. Turn off the apparatus. The flames should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.
- Be sure that this test is not conducted in an area where there may be vapors present from highly flammable substances, such as ether.
- Absolutely no flammable materials should be in the hood with the flash point apparatus.
- Fire extinguisher will be located near the flash point apparatus.
- Be cautious with samples that exhibit flammable characteristics (e.g., smell like solvent) when conducting this test.
- Keep the tester under a hood, but contained in such a way that no drafts affect the vapors at the top of the cup.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Decane or n-Decane	Flammable Irritant	500 ppm-TWA	**Inhalation:** Moderately irritating to the respiratory tract, especially in high concentrations. Coughing, mild inflammation and possible dizziness or other narcotic effects can be expected. **Ingestion:** Mildly irritating to the gastro-intestinal tract, causing abdominal spasms, nausea, vomiting (aspiration hazard!) and diarrhea. **Skin Contact:** Mild irritant and may cause inflammation and skin cracking due to defatting action. **Eye Contact:** Mild irritant, especially if splashed into the eye. Inflammation, tearing, pain can result. **Chronic Exposure:** Continued or repeated skin contact may cause dermatitis. **Aggravation of Pre-existing Conditions:** No information found.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Herzog Model HFP 339, Automated Closed-Cup Flash Point Tester

For safety and function, the apparatus is set up inside a clear plastic portable draft-eliminating hood that is set within a continuously operating fume hood. No drafts may affect the vapors in the test cup.

The thermometer is specific to the instrument and must be purchased from the vendor. It is calibrated annually against an NIST-certified thermometer at 2 temperatures that bracket the range of use. The recommended testing temperatures are 80° F and 177° F. The tester is equipped with a barometric pressure sensor and automatically corrects the observed flash point.

4.2 Cleveland Open Cup Flash Point Tester

The tester must be set up in a clear plastic portable draft-eliminating hood that is set within a continuously operating fume hood, thus contained in such a way that no drafts affect the vapors at the top of the cup. An ASTM thermometer specifically for Cleveland Open Cup flash point testing is required.

4.3 Barometer, Fisher Scientific 14-648-51

The barometer is mounted on the wall and must be read with the mercury at eye level. First adjust the pointer so that it just indents the surface of the mercury pool, then read the mmHg on the right-side scale remembering that the meniscus is inverted compared with a water meniscus. Record the pressure in the IMB book with the open cup flash point batch.

5.0 REAGENTS AND STANDARDS

5.1 n-Decane

Purchased from a chemical vendor (J.T. Baker catalog # G143-07 or equivalent). (closed cup flash point 127°F).

- Life of Reagent: 5-years
- Storage Requirements: **FLAMMABLE**, store in a flammables cabinet

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit[1]
Laboratory Control Sample (*n-Decane* Standard)	Each Closed Cup batch	127 ± 4 °F (123-131°F)
Matrix Duplicate (DU)	1 in 20 samples	± 4 °F difference for closed cup Method A ± 9 °F difference for closed cup Method B ± 18 °F difference for open cup

[1] The limits are taken from precision limits (95% confidence) given in the ASTM standards, 1985 versions.

7.2 Sample Preservation and Storage

Holding time and preservation techniques are not specified in the referenced methods. Sample containers should be air tight and glass or metal containers are recommended for organic solvents and oils.

7.3 Sample Preparation

7.3.1 Sample Size

Approximately 70 mL is required to fill either flash point cup to the level indicated by the filling mark inside of the cup.

7.3.2 Preparation

Mix the sample well. Viscous samples may be warmed but only enough to mix and dispense the sample. Cool the sample to at least 20°F below the expected flash point for closed cup testing and 50°F below the expected flash point for open cup testing, if possible.

Note: Certain clients have requested that in order for solid samples to be stirred during closed-cup analysis, the samples can be mixed with a minimum amount of mineral oil. There has been no study done at TestAmerica Chicago to determine the effect of this protocol on the flash point result on any matrix.

7.4 Calibration / Standardization

The thermometer for each instrument must be calibrated against an NIST thermometer upon receipt and annually thereafter.

7.5 Preventive Maintenance

- Frequently examine the Pensky-Martens stirring mechanism for weakness due to corrosion.
- The cup should be completely cleaned and DRIED before each test. Clean the cup with soap and water, solvents, and steel wool. Be sure to dry and remove solvents or a false positive could result.
- It is **imperative** to examine the Herzog electrical igniter prior to operation for signs of mechanical or physical damage. A damaged igniter could result in a no-flash on a pretest of a flammable sample, resulting in a high expected flash point to be entered. In that event, it is possible for the unit to over heat the sample, **and a fire could result**.
- Do not clean the Herzog keyboard with any kind of solvent.

7.6 Sample Analysis

7.6.1 Set-Up and Operation – Herzog Closed Cup Analyzer

NOTE: The instrument can be programmed to report results in °F or °C. TestAmerica Chicago reports flash point in °F.

7.6.1.1 Fill the cup to the mark.

7.6.1.2 Place and lock the lid on the cup and insert the thermometer. Insert the thermometer directly into any solid sample. Also, insert the thermal detector and attach the drive rod.

7.6.1.3 If a liquid sample is being analyzed, attach the stirring mechanism. The instrument will automatically stir the sample at the appropriate rate if the stirring mechanism is assembled. **Do not attempt to stir soil samples.**

NOTE: Refer to the instrument manual for more detailed instructions, if needed. The "Menu" button toggles between various options, including "Diagnostics" and "Ready".

7.6.1.4 The display should read "Ready". If not, keep pushing the "Menu" key until "Ready" appears, then press "Start". You will be asked to enter a sample number, which is optional, since TestAmerica Chicago doesn't currently export data to LIMS from this instrument.

7.6.1.5 Press "Start" again. You will be asked to enter program 3 or 4. Choose program 3, ASTM Method A for the n-Decane standard and liquid samples, or program 4, ASTM Method B, for solid samples.

7.6.1.6 Press "Start" again. You will be asked to enter the expected flash point. For the n-Decane standard, enter 127° F as the expected flash point. For most samples, enter 140° F as the expected flash point. If a sample apparently contains a solvent, a lower temperature may be chosen. The ignitor is applied beginning 41° F below the expected flash point and continues for 36° F beyond the set point, unless a flash is detected. If 140° F is used as the expected flash point, the test will begin at 99° F and end at 176° F or when a flash point is detected, whichever is lower.

Note: 140° F is the SW-846 ignitability hazard point.

7.6.1.7 After entering the expected flash point, press "Start" once again. You will be asked whether or not to start the test. Enter "Yes". After a brief self check, the instrument will automatically apply the ignition coil to the sample every 2° F. The rate of temperature rise and the stirring speed are also pre-programmed.

7.6.1.8 A buzzer will sound if a flash point is detected and the flash point will be displayed on the screen. If no flash point was detected up to the high temperature of 176° F, the screen will read "error". The cooling cycle will automatically begin when the test is over.

Note: If possible, choose a sample that flashes as the duplicate for the batch, as a means of confirmation as well as a measure of precision.

7.6.2 Cleveland Open Cup

7.6.2.1 Place the ASTM thermometer into the sample cup so that the bottom of the bulb is ¼ inch from the bottom of the cup.

7.6.2.2 Fill the clean, dry cup to the line with sample. The method was written for liquid samples, but if a solid is being analyzed, smooth the surface as much as possible to the sample line.

7.6.2.3 Light the test flame and adjust it to 3.2-4.8 mm (1/8 to 3/16 inches) diameter.

7.6.2.4 Turn up the heater control dial so that the sample temperature rises 25-30 °F per minute. When the temperature reaches approximately 100 °F below the expected flash (or at 100 °F for most non-solvent-containing samples) decrease the heat so that the temperature of the sample rises 10 +/- 1 °F per minute.

7.6.2.5 Starting at 50 °F below the expected flash (no lower than 130°F for most samples), apply the flame every 5 °F until the sample flashes, boils, or reaches 200 °F. With a smooth, continuous motion, pass the flame over the sample so that it takes approximately 1 second to cross over the cup. Cross over in the opposite direction the next time.

7.6.2.6 The observed flash point is the temperature when any point on the surface of the sample flashes, but do not confuse a flash with a bluish halo that may form around the flame. Correct the observed flash point for the barometric pressure by the following equation:

Flash point in °F = Observed FP in °F + 0.06(760 - mmHg BP)

See section 4.3 for details about the barometer.

7.7 Documentation

7.7.1 Analysis Logbook

Record the sample #, a brief sample description (such as "yellow, turbid solvent" or "gray, heavy, wet clay"), the temperature at which the test was begun, and the observed flash point, in the IMB data book (Attachment 1). Fill out all appropriate header information in the data book. This runlog must be completed for each day's analysis. Enter the results into TALs LIMS.

8.0 QUALITY CONTROL

8.1 QC Summary

A DU and LCS are analyzed with every batch of 20 or fewer samples for closed cup, but no LCS is done for open cup.

Note: TALS is not programmed to calculate or evaluate a +/- acceptance limit. Therefore, the flash point QC is not entered into TALS.

8.2 Corrective Actions

When an out of control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out of control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel, or other experienced staff if he/she is uncertain of the cause of the out of control situation. The test must not be resumed until the source of the problem and an in-control status is attained. All samples associated with the out of control situation should be reanalyzed. Out of control data must never be released without approval of the supervisor, QA personnel or the lab manager.

Listed below are steps that must be taken when an out of control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist;
- receive approval (signature) of the supervisor, QA personnel, or the Laboratory Manager prior to the release of any analytical data associated with the problem.

Suggested Actions to specific out of control situations:

8.2.1 Laboratory Control Standards (LCS)

- reanalyze the LCS to verify that an out-of-control situation exists;
- determine the source of error within the preparation procedure, correct the problem and repeat the sample set. (Sources of error could be the wrong expected flash point having been entered or contamination with cleaning solvents.)

8.2.2 Matrix Duplicate (DU)

- the sample must be analyzed a third time;
- report the 2 closer results as the sample result and duplicate result;
- write an NCM.

9.0 Reporting Results

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Transcription errors

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Samples that flash during this procedure will enter the "flammable liquid" wastestream.
- Samples that do not flash during this procedure will enter the "waste water" wastestream or non-hazardous solid waste containers.
- Expired chemicals and single component standards will be turned over to the EHSC or waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.

13.0 ATTACHMENTS

Attachment 1: Example: Analysis Log
Attachment 2: Data Review Checklist

14.0 REVISION HISTORY

- Revision 16 updated on 11/08/12
- Annual Review
- Added to section 4.3 to clarify the barometer use

Attachment 1.

Example: Analysis Log
(011-001)

TestAmerica Chicago

Miscellaneous (IMB)

Parameter: _____

LIMS Test Method: _____ LIMs Batch #: _____

Reporting Limit: _____

Drinking Water Set (Circle): YES NO

Prep Time: _____ Start _____ End

Analysis Time: _____ Start _____ End

Page #: _____

Book #: _____

Instrument: _____

Conditions: _____

Calculation: _____

Stds. / QC / Sample #		Results	Units	% Rec RPD

Standard Traceability:	Reagent Traceability:	Eppendorf Traceability:

Note: Working Standards are prepared daily from the noted Stock Solutions.

Analyst Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

CHI-22-12-006/E-9/07

(011-001)

Attachment 2.

Example: Data Review Checklist
(012-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST

Test _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Is the Batch Information screen complete? Analyst correct? ____ Start/End times correct? ____ Instrument Code entered? ____ Are Batch Notes complete? (i.e. reagents, filters, temperatures, etc....) ____			
2) Was Data Imported ____ Manually entered ____ Balance Interface Used ____			
3) Samples & all QC in order as analyzed? (All readings must be present in sequence.)			
4) Sample List Screen Date(s)/Time(s) correct			
5) Standard Codes present and Amount Spiked correct?			
6) Dilution factors all present and correct?			
7) Are correct Sample ID's used? ____ Data entries match raw data? ____ Are all samples designated with a Blue P? ____			
8) Are correct QC ID's used? ____ Data entries match raw data? ____ Are all QC designated with a Blue P? ____			
9) Is QC correctly linked to each sample?			
10) Is all QC calculated and are correct flags applied?			
11) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; ____ Holding Time missed or Manual Integration required? ____			NCM # _____ Approved By: _____ Initials
Raw Data: 1) Are calibration criteria met? $r \geq 0.995$; $\lvert$y-intercept$\rvert$ < RL; (<1/2 RL DoD QSM)			
2) Is AD Batch # clearly identified?			
3) Are manual calculations and final results clearly shown?			
4) Are all errors crossed out with single line & initialed and dated? Are all unused portions of the pg. Z'd out?			
5) Is unreported data clearly identified with reason & initialed and dated?			
6) Is data signed & dated by analyst & reviewer?			

(012-001)

TITLE: WET CHEMISTRY pH

Approvals (Signature/Date):

Carla Bonner 8/29/13 Supervisor, Wet Chemistry	Diane L. Harper 8/28/13 Inorganics Manager
Terese A. Preston 8/28/13 Quality Assurance Manager	Mark Storm 8/28/13 Env. Health & Safety Coor.
Michael J. Healy 8/28/13 Laboratory Director	

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TITLE: WET CHEMISTRY pH

Approvals (Signature/Date):

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Terese A. Preston — 8/28/13 Quality Assurance Manager	Mark Storm — 8/28/13 Env. Health & Safety Coor.
Michael J. Healy — 8/28/13 Laboratory Director	

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1.0 SCOPE / APPLICATION

This Standard operating Procedure (SOP) is used to determine the pH of laboratory samples by the appropriate SW-846 or equivalent method. This SOP was written using SW-846 Methods 9040B, 9040C, 9045C, 9045D and 9041A as primary references.

Standard Methods 20th Ed., 4500-H^{+}-00 is equivalent to SW-846 Method 9040B and 9040C. There are no equivalent methods for SW-846 methods 9045C, 9045D or 9041A.

There may be several references to the equivalent method EPA 600/4-79-20 Method 150.1 throughout this SOP. As of March 12, 2007, the USEPA has withdrawn this method from the Federal Register. There will be a transition period in which the method reference is removed from the laboratory, agency, and permit documentation. Regardless, TestAmerica Chicago will remain in compliance with approved methods and regulations.

On occasion, clients request slight modifications to this SOP. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

Not Applicable.

1.1.2 Reporting Limits

The reporting limit for pH is statistically determined from LCS (7.0 buffer) data as ± the true value. Routine reporting limits for samples are reported as ± 0.1 SU for water samples and ± 0.2 SU for soil samples.

1.1.3 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

Samples containing < 20% liquid by volume must be analyzed by SW 9045C/9045D. Aqueous samples require no special preparation step.

For SW 9040B/9040C and 9045C/9045D, a pH meter equipped with a pH electrode and a temperature compensator probe is calibrated. The calibration is verified with alternate-source buffers. Two LCSs, the aqueous samples, and the aqueous phases of solid sample preparations are then analyzed. After every 10 readings, the calibration verification buffer is read.

SW 9041A is used for difficult matrices or when requested specifically by the client. It is **NOT** applicable for the determination of the RCRA characteristic of corrosivity. The pH of a sample is first taken with verified broad-range pH paper test strips. Verified narrow range paper strips are then used to determine the pH more accurately.

2.0 INTERFERENCES

Soaps, oily matter and suspended solids may coat the glass electrode and cause a sluggish response. However, these materials may not be removed to increase precision because some may contribute an important part of the acid-consuming property.

Certain wastes may mask the color change necessary to accurately read paper pHs. Addition of acid and base to aliquots of the waste may detect the masking.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

None.

3.2 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

4.0 EQUIPMENT AND SUPPLIES

- pH meter
- Combination pH electrode compatible with meter
- temperature compensator electrode
- analytical balance, capable of weighing 0.1 gram
- stir plate and bars
- 50 mL disposable beakers
- narrow range pH test strips
- broad range pH test strips
- small weigh boats

5.0 REAGENTS AND STANDARDS

Use only reagent grade or better chemicals that conform to ACS specifications.

5.1 Calibration Standards

pH Buffers ~2.0; 4.0; 7.0; 10.0; and 12.0. When entering true pH values into laboratory LIMS, record the true value of the buffer at the assumed laboratory temperature of 21°C.

Purchased through a chemical supplier.

- Life of Reagent: 1 year or manufacturer's recommendation
- Storage Requirements: None

5.2 Laboratory Control Sample (LCS)
Initial Calibration Verification (ICV) Standard
Continuing Calibration Verification (CCV) Standard

Low Range ICV/CCV	pH Buffer 4.0
LCS	pH Buffer 7.0
High Range ICV/CCV	pH Buffer 10.0

Purchased from an alternate manufacturer than the Calibration Standards.

- Life of Reagent: 1 year or manufacturer's recommendation
- Storage Requirements: None

5.3 Hydrochloric Acid, 1/4 dilution

Place 750 mLs deionized (DI) water into a 1 L beaker. Under a hood, carefully add 250 mLs concentrated hydrochloric acid (HCl). Mix well.

- Life of Reagent: 1 year
- Storage Requirements: None

5.4 Sodium Hydroxide, ~0.1 N

Place ~500 mLs DI water into a 1-Liter beaker. While stirring under a hood, carefully add 4.0 grams sodium hydroxide (NaOH) pellets. Dilute to a 1 liter.

- Life of Reagent: 1 year
- Storage Requirements: None

6.0 CALIBRATION (NON-DAILY)

Not Applicable.

7.0 PROCEDURES

7.1 Quality Control Checks

Quality Controls	Frequency	Control Limit
pH 4.0, 7.0 & 10.0 (Cal.) or other standards as needed to bracket expected sample pHs.	prior to samples	± 0.10
pH 4.0 (ICV)	prior to samples	± 0.10
pH 7.0 (LCS & LCSD)	every run	± 0.10
pH 4.0 & 10.0 (CCV)	Alternate every 10 readings and at the end of each run	± 0.10
Matrix Duplicate(DU)	All waters, 1 in 20 or fewer soils	± 0.20

7.2 Sample Preservation and Storage

In order to be in compliance with the EPA holding time, aqueous samples must be analyzed on site, immediately after collection. The laboratory analyzes samples as soon as possible on the day of receipt, but all sample results are flagged "HF" which identifies it as a field test that was delayed for laboratory analysis.

There is no established holding time for soils or solids, although TALS defaults to 7 days from collection. pH sample aliquots are not preserved, except for refrigeration in transport.

7.3 Sample Preparation

7.3.1 Sample Size

Matrix	Sample Size
Waters	Minimum of 25 mLs
Solids	20 grams

7.3.2 Soils and Solids containing < 20% liquid by volume only, (SW 9045C, 9045D)

To 20 grams of each soil or non-soil waste in a 50 mL beaker, add 20 mLs DI water. Stir the suspensions for 5 minutes then allow the suspensions to settle for approximately 1 hour before analysis. If the water is absorbed by the sample, add additional DI water until a sufficient aqueous phase is present, and note the addition. For non-soil waste samples, let the mixture settle for at least 15 minutes, then filter or centrifuge out the solids if necessary and analyze the aqueous phase.

7.4 Calibration / Standardization

7.4.1 All buffers and samples should be approximately room temperature. Pour the standardizing buffers, LCS and verification buffers, and all water samples into 50 mL beakers.

Note: All buffers must be freshly poured daily. Keep 500 mL source bottles tightly capped and at room temperature.

7.4.2 Using the 4.0, 7.0, and 10.0 buffers, calibrate the meter according to the instructions. Gently stir all buffers while taking the readings. Record the pHs in the data book or directly into an ADII batch. See Section 7.1 for acceptance limits.

7.4.3 Record the slope (%) in the data book, acceptance criteria is 92% to 102%.

7.4.4 After calibration read the alternate source 7.0 buffer in duplicate as LCSs. At that point and then after every 10 or fewer readings, alternately read the 4.0 and 10.0 buffers as continuing calibration verification. Again, all buffers should be read while being gently stirred.

7.4.5 When measuring the pH for waste characterization (Corrosivity), samples with pHs above 11.0 must be brought to an actual temperature of 25 $\pm$ 1° C.

NOTE: For corrosivity characterization, a pH ~2.0 buffer must be used for acidic waste and a pH ~12.0 buffer must be used for caustic waste.

7.5 Preventive Maintenance

7.5.1 pH paper test strips should be kept dry and reasonably tightly covered.

7.5.2 The probes are rinsed with DI water in between samples. When analyzing samples, the probes should be cleaned thoroughly with a soft tissue to insure an accurate reading. Oily residues may be removed be gently scrubbing in hot, soapy water. Rinse thoroughly afterward.

7.5.3 See manufacturer's recommendations for proper maintenance and storage of the electrodes.

7.5.4 Verify the temperature sensing of the ATC probe annually against an NIST traceable thermometer and note any correction factor. This factor must be applied to all following data.

7.5.5 Annually, verify the function of the ATC probe by calibrating the meter, then placing the pH and ATC electrodes into pH 7.0 buffer that has been cooled to < 10 $^{\circ}$ C. Record the pH and temperature. Slowly raise the temperature to ~25° C and ~ 35° C, again recording the temperature and pH. The pH reading should remain stable. If not, contact the manufacturer for instructions immediately.

7.6 Sample Analysis

7.6.1 Electrode Method

7.6.1.1 Read and record the pHs and temperatures of all the samples. Stir the water samples while taking the readings, but only lower the electrodes into the aqueous phase of the soil/solid preparations from Section 7.3. Each water sample must be read in duplicate.

7.6.2 Paper Method

7.6.2.1 Each batch of narrow range paper must be calibrated against purchased pH buffers prior to use. Dip the appropriate test strip into the pH 10.0, pH 7.0, and pH 4.0 calibrating buffers. The paper reading must be within 0.5 pH units of the known value of the buffer in all cases.

7.6.2.2 Place 4, small, representative aliquots of each sample into small weigh boats.

7.6.2.3 Determine the approximate pH of the sample by applying a piece of the broad range paper to the first aliquot. Record the result and the color change of the paper.

7.6.2.4 Apply 2 separate pieces of the appropriate narrow range test strip to different locations of the second aliquot. This is considered a duplicate and all samples must be tested in duplicate. Record the pHs, the range of the pH paper, and the observed color changes.

7.6.2.5 Under a hood, add dilute acid (Rgt.5.3) dropwise to the 3rd aliquot until a color change is observed with the narrow range paper. Record that a color change was observed.

7.6.2.6 Under a hood, add dilute base (Rgt.5.4) dropwise to the 4th aliquot until a color change is observed with the narrow range paper. Record that a color change was observed.

NOTE: The observance of color changes in steps 7.6.2.5 and 7.6.2.6 is a strong indication that no matrix interferences have occurred.

7.7 Documentation

7.7.1 Analysis Logbook

Sample and pH standard analysis is documented in the dedicated logbook and subsequently transferred into TALs. Alternatively, the results may be entered directly, either manually or electronically, into an ADII batch. Care must be taken either way to identify all buffer source IDs, sample anomalies, etc.

7.7.2 Reporting Results

Enter the pH set into TALS, completing and reviewing all the entries as required by the data entry checklist. Calculate and set the status to first level review, ensuring that the QC is correct, and that all the results are reporting as expected. Write any necessary Non-Conformance Memos (NCMs). Submit a completed and signed checklist along with the TALS print-outs and the signed data book, if applicable, to a qualified reviewer. The reviewer will verify the completeness and correctness of the data batch, and set the batch status to second level reviewed.

7.7.3 Data Review

Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 2) and sets the batch status in LIMs to 1st level review. The second level or peer review of the data is conducted by another individual who has been trained on the review process. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to 2nd level review. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Raw data indicating some type of contamination or poor technique
- Transcription errors

8.0 QUALITY CONTROL

8.1 QC Summary

8.1.1 Two LCSs will be included in each laboratory run and are examined to determine both precision and accuracy.

8.2 Corrective Actions

When an out-of-control situation occurs, the analysts must use his/her best analytical judgment and available resources to determine the corrective action to be taken. The out-of-control situation may be caused by more than one variable. The analyst should seek the assistance of his/her immediate supervisor, QA personnel or other experienced staff if he/she is uncertain of the cause of the out-of-control situation. The test must not be resumed until the source of the problem and an in-control status is attained.

All samples associated with the out-of-control situation should be reanalyzed. Out-of-control data must never be released without approval of the supervisor, Project Manager, QA personnel or the Lab Director.

Listed below are steps that MUST be taken when an out-of-control situation occurs:

- demonstrate that all the problems creating the out of control situation were addressed;
- document the problem and the action which was taken to correct the problem on the data review checklist;
- document on the checklist that an in-control has been achieved; and
- receive approval (signature) of the Supervisor, Inorganics Manager, Project Manger, QA personnel, or the Laboratory Director prior to the release of any analytical data associated with the problem.

Suggested Actions to specific out-of-control situations:

8.2.1 Initial Calibration Verification (ICV)

- repeat ICV to verify proper reading;
- pour new ICV from original stock;
- recalibrate, reanalyze;
- pour fresh buffers if necessary.

8.2.2 Laboratory Control Samples (LCSs)

- re-read LCS to verify that an out-of-control situation exists;
- determine the source of the error, correct the problem and repeat the sample set. (Sources of contamination could be the electrode, the LCS stock solution, glassware, or the preparation area.)

Precision: LCS and LCSD must meet the control limits of $\pm$ 0.10 pH. If these criteria are not met, then see your supervisor for proper corrective action.

8.2.3 Matrix Duplicate (DU)

- the sample must be reprocessed and reanalyzed;
- if the reanalysis is within the control limits, the second value is reported;
- document that the corrective action was done on the data checklist for the set.

8.2.4 Continuing Calibration Verification (CCV)

- repeat CCV;
- pour fresh buffer if necessary;
- recalibrate and repeat all samples since the previous in control CCV;
- never dispose of any samples until you are sure that all QC are within their designated control limits.

9.0 DATA ANALYSIS AND CALCULATIONS

Percent recoveries and RPDs are not applicable to pH. Control limits are in ± actual difference from the known or original. Note: TALS will not calculate and report the actual difference.

10.0 POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to UP-WM-001.

The following waste streams are produced when this method is carried out.

- Alkaline sample waste generated by the analysis will be collect in approved containers and poured into the Carboy labeled "Corrosive Liquid " waste using a funnel to reduce splashing.
- Acidic sample waste generated by the analysis will be collect in approved containers and poured into the Carboy labeled "Corrosive Liquid" waste using a funnel to reduce splashing.
- When expired, the buffer solutions utilized in the analysis will be turned into the waste technician for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to SOP UP-QA-014, *Laboratory Training: Skills and Mechanisms* for additional details.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Attachment 1. Example: Analysis Logbook
Attachment 2: Example: Data Review Checklist

14.0 REVISION HISTORY

- Revision 65 updated on 08/28/13
- Annual Review
- SOP Change Form updated to sections 1.1.2; 7.4.1; 7.6.2.5; 7.6.2.6
- Section 7.4.3 was updated with the pH Meter slope criteria.

Attachment 1.

Example: Analysis Logbook
(011-001)

TestAmerica Chicago
pH

LIMS Batch No.:_____ Page No. _____

Method:
a. SW 9040B d. SW 9045D
b. SW 9040C e. SM 4500H^{+} 00
c. SW 9041A f. ICORP[1]
d. SW 9045C

[1]Corrosivity: <4 or >10 pH. Consult the SOP for additional requirements.
[2] Calibration Acceptance Criteria: ± 0.1 SU
[3] Slope Acceptance Criteria: 92% to 102%

Instrument: _____
NCM No.: _____

Calibration[2]

	Source ID#	pH	Temp °C	
Standard				
Standard				**Slope[3]**
Standard				
ICV				
7.0 LCS				**Diff.**
7.0 LCD				

	Time	Sample #	Matrix	Method #	pH Result	Temp °C	pH Difference Comment
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
		_____	CCV _____				
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
		_____	CCV _____				
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
		_____	CCV _____				

Analyst Signature:_____ Date:_____ Time:_____

Reviewer Signature:_____ Date:_____

(011-001)

Attachment 2.

Example: Data Review Checklist
(012-001)

TestAmerica Chicago
INORGANICS – LIMS DATA REVIEW CHECKLIST

Test _____ **Analytical Batch#** _____

Prep Batch # _____
(File by analytical batch #)

Analyst / Primary Reviewer: _____ 1st Level Review Date: _____

Secondary Reviewer: _____ 2nd Level Review Date: _____

	PRI REV	SEC REV	COMMENTS
1) Is the Batch Information screen complete? Analyst correct? _____ Start/End times correct? _____ Instrument Code entered? _____ Are Batch Notes complete? (i.e. reagents, filters, temperatures, etc….) _____			
2) Was Data Imported _____ Manually entered _____ Balance Interface Used _____			
3) Samples & all QC in order as analyzed? (All readings must be present in sequence.)			
4) Sample List Screen Date(s)/Time(s) correct			
5) Standard Codes present and Amount Spiked correct?			
6) Dilution factors all present and correct?			
7) Are correct Sample ID's used? _____ Data entries match raw data? _____ Are all samples designated with a Blue P? _____			
8) Are correct QC ID's used? _____ Data entries match raw data? _____ Are all QC designated with a Blue P? _____			
9) Is QC correctly linked to each sample?			
10) Is all QC calculated and are correct flags applied?			
11) Is an NCM needed? ICV, MB, LCS, LCSD, DU, MS, MSD, RPD out; _____ Holding Time missed or Manual Integration required? _____			NCM # _____ Approved By: _____Initials
Raw Data: 1) Are calibration criteria met? $r \geq 0.995$; $\lvert$y-intercept$\rvert$ < RL; (<1/2 RL DoD QSM)			
2) Is AD Batch # clearly identified?			
3) Are manual calculations and final results clearly shown?			
4) Are all errors crossed out with single line & initialed and dated? Are all unused portions of the pg. Z'd out?			
5) Is unreported data clearly identified with reason & initialed and dated?			
6) Is data signed & dated by analyst & reviewer?			

(012-001)

Appendix C
Example Chain of Custody

USEPA CLP Organics COC (REGION COPY)

DateShipped:
CarrierName: FedEx
AirbillNo: 8273 6007 7665

CHAIN OF CUSTODY RECORD

OMC GW Monitoring Mar2012/WI
Case #: 42320
Cooler #:

No: 5-030712-144500-0021

Lab: PEL Laboratories
Lab Contact: John Heyman
Lab Phone: 813-888-9507

Organic Sample #	Matrix/Sampler	Coll. Method	Analysis/Turnaround	Tag/Preservative/Bottles	Station Location	Colle...	...rganic ...ple #	Sample Type
E4PX5	Ground Water/ CH2M HILL	Grab	PCBs(21), PCBs(21)	5-31400 (4 C), 5-31401 (4 C) (2)	OMC-MW528D		...X5	Field Sample
E4PX6	Ground Water/ CH2M HILL	Grab	PCBs(21), PCBs(21)	5-31408 (4 C), 5-31409 (4 C) (2)	OMC-...			Field Sample
E4PX8	Ground Water/ CH2M HILL	Grab	PCBs(21), PCBs(21)	5-31424 (4 C), 5-3142... (4 C) (2)				Field Sample
E4PX9	Ground Water/ CH2M HILL	Grab	PCBs(21), PCBs(21)	5-31432 (4 ...				...ld Sample

...or Project Code | Sample Number | Month/Day/Year | Time | DESIGNATE Comp. | Grab

...tion Number and Location | Samplers (signatures)

Matrix: Rema...

PRESERVATIVE: ...SO4 ☐ ICE ☐ HCL ☐ HNO3 ☐ NaOH ☐ Other ☐

ANALYSES: VOA, ABN, PEST/PCB, METALS, CYANIDE, ...itrite

Station Loc ID: OMC-MW11D
Sample No.: E4PW5
Analysis: CLP TCL Volatiles
Tag No.: 31311

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION V
OFFICIAL SEAL
No 195127

Special Instructions: CUSTODY SEALS 195127, 195128

...hain of Custody #

Analysis Key: PCBs=CLP Aroclors

Items/Reason	Relinquished by	Date	Received	Date	Received by	Date	Time
	Joe Sampler	5/4/2012					

Lab Sample Number

Appendix D
CMA Trident Probe Standard Operating Procedure

TRIDENT PROBE STANDARD OPERATING PROCEDURES

TECHNOLOGY DESCRIPTION

The Trident probe is a direct-push, integrated temperature sensor, conductivity sensor, grain-size sensor and porewater sampler developed to screen sites for areas where groundwater may be discharging to a surface water body (Chadwick et al., 2003; Figure 1). Differences in observed conductivity and temperature indicate areas where groundwater discharge is occurring. The integral porewater sampler can be used to rapidly confirm the presence of groundwater constituents and map the subsurface distribution of contaminants of concern.

Trident Conductivity/Temperature Sensors

The subsurface temperature sensor consists of a ruggedized digital oceanographic thermometer embedded near the tip of a 90 cm long stainless-steel probe (Figure 1). The sensor has a measurement range of -5 to +45 °C at an accuracy of <0.1 °C, and a resolution of 0.001 °C. The sensor response time is about 60 s. The subsurface conductivity probe utilizes a small diameter, stainless steel, AC-excitation 3-electrode sensor, installed at the tip of the same 90 cm probe that houses the temperature sensor (Figure 1). The conductivity sensor has a range of 0 to 80 mS/cm, an accuracy of <2% of the calibrated range, and a resolution of 0.01 mS/cm. The subsurface probe is used to measure the bulk conductivity signal which varies primarily as a function of changes in salinity, and secondarily as a function of clay content and porosity. A reference conductivity and temperature sensor also is mounted on the instrument frame to provide a direct comparison of the overlying surface water conditions with the interstitial water conditions (Figure 1). For the temperature sensor, areas of groundwater seepage may appear either as warm or cold contrast to the surface water depending on the seasonal and site characteristics. For the conductivity sensor, areas of likely groundwater seepage are generally associated with low conductivity in coastal areas where fresh groundwater is discharging to seawater, but may be associated with high conductivity in rivers and lakes

where the groundwater often has higher total dissolved solids relative to the surface water.

Liquid-Tip Conductivity Sensor

The subsurface conductivity probe can also be used in the “liquid tip” configuration in which the sensor is housed within a secondary screen and responds only to the porewater conductivity (Chadwick et al., 2007; Figure 2). The liquid tip configuration uses a small diameter sampling tube to purge the conductivity cell until the readings stabilize (generally <100 ml; Figure 3). The data are then recorded in the same manner as with the bulk conductivity sensor.

Data Acquisition System

Trident sensors are coupled through an underwater connecter and cable to a deck unit that integrates the probe and reference temperature, conductivity and SED-FSP signals with the signal from a Global Positioning System (GPS) sensor mounted on the top of the push-pole (Figure 1). The GPS is a Garmin model 17 with a stated accuracy of <15 m in standard mode, and <3 m in Wide-Area Augmentation System (WAAS) mode. The integrated data stream from the deck unit is sent to a laptop via RS-232. The laptop is used to apply calibration and temperature corrections to the signals, and record and display the results.

Porewater Sampler

The water-sampling probe allows interstitial waters to be extracted from the sediment at selected depths up to about 90 cm below the sediment water interface. Porewater is collected by a low-flow peristaltic pump extraction through a small-diameter, Teflon-coated, stainless steel probe (Figure 4). The probes consist of a length of 9.5 mm diameter stainless steel tubing fitted with a solid, removable point. On the side of the tube near the tip there is a sample port consisting of a hole covered by a small mesh size (250 μm) stainless steel screen. The screen section is easily removable for cleaning or replacement if required (Figure 4). The porewater sampler can also be configured with a secondary screen with a sand-pack to provide a pre-filter for the sampling and to minimize clogging of the sampler (Figure 5). This secondary screen is installed over the

outside of the probe, and the void between the probe and the screen is packed with pre-cleaned sand. Multiple probes can be used to together to further increase surface area, enhance sampling rate, and minimize potential clogging.

FIELD SAMPLING PROCEDURE

A Trident survey is conducted by inserting the probe into the seabed (seabed is used here to mean the bottom of the ocean, estuary, or bay) from a boat or by wading. In operation, the Trident probe can be deployed in several ways depending primarily on the depth of the site. In water of moderate depths (1-10 m), the probe is easily deployed from a small boat using the push rod (Figure 6). It is important that the boat be well anchored to minimize lateral loading on the probe during the insertion. In deeper water (>10 m), the probe can be deployed by diver, or can be attached to a landing frame (Figure 6). In very shallow water (<1 m) the probe can be installed by wading.

Conductivity and Temperature Sampling

Once on station with the probe inserted, data is collected from the conductivity and temperature sensors using the TridentTalk software. If the liquid tip sensor is being used, the cell must be purged prior to data collection. The TridentTalk software provides a display of the probe and reference temperature and conductivity signals, along with the GPS position. The software also automatically calculates and displays the probe vs. reference temperature and conductivity contrast. Once the sensor readings have stabilized, the data is recorded by activating the “Log current data” button on the TridentTalk display. The data can then be reviewed in numeric format, or displayed spatially using the AGIS graphical information system software. The spatial AGIS display provides a capability for rapidly evaluating the most likely areas of groundwater discharge based on temperature and conductivity contrast. Once data recording is completed, the probe is retrieved. If the liquid tip sensor is being used, the sensor cell must be cleaned between stations by removing any accumulated particles and rinsing the cell with appropriate solution (generally surface water). A typical shoreline sampling grid for a Trident sensor survey is shown in Figure 7. Typical results for a liquid-tip sensor survey are shown in Figure 8.

Porewater Sampling

Once on station with the porewater probe inserted, the probe and sampling tube is purged of ~3 volumes using the sampling pump. The pump should be run at a low enough rate to avoid over pressure and clogging or excessive vacuum bubbles in the sampling line. With the sampler purged, the porewater samples can then be collected in accordance with the volume and quality control requirements of the project. Once the sampling is complete, the probe is retrieve and decontaminated in accordance with project requirements prior to sample collection at the next station. Typical results for a shoreline porewater sampling survey are shown in Figure 9.

REFERENCES

Chadwick, D.B., J. Groves, C. Smith, and R. Paulsen. 2003. Hardware description and sampling protocols for the Trident Probe and UltraSeep system: Technologies to evaluate contaminant transfer between groundwater and surface water. Technical Report #1902, SSC San Diego, United States Navy.

Chadwick, D.B., J. Guerrero, G. Rosen, J. Groves, C. Smith, R. Paulsen, A. Burton, and M. Greenberg, 2007. The Trident Probe capabilities and applications for identifying and mapping groundwater discharge zones. Proceedings of SETAC North America 28th Annual Meeting, Milwaukee, WI.

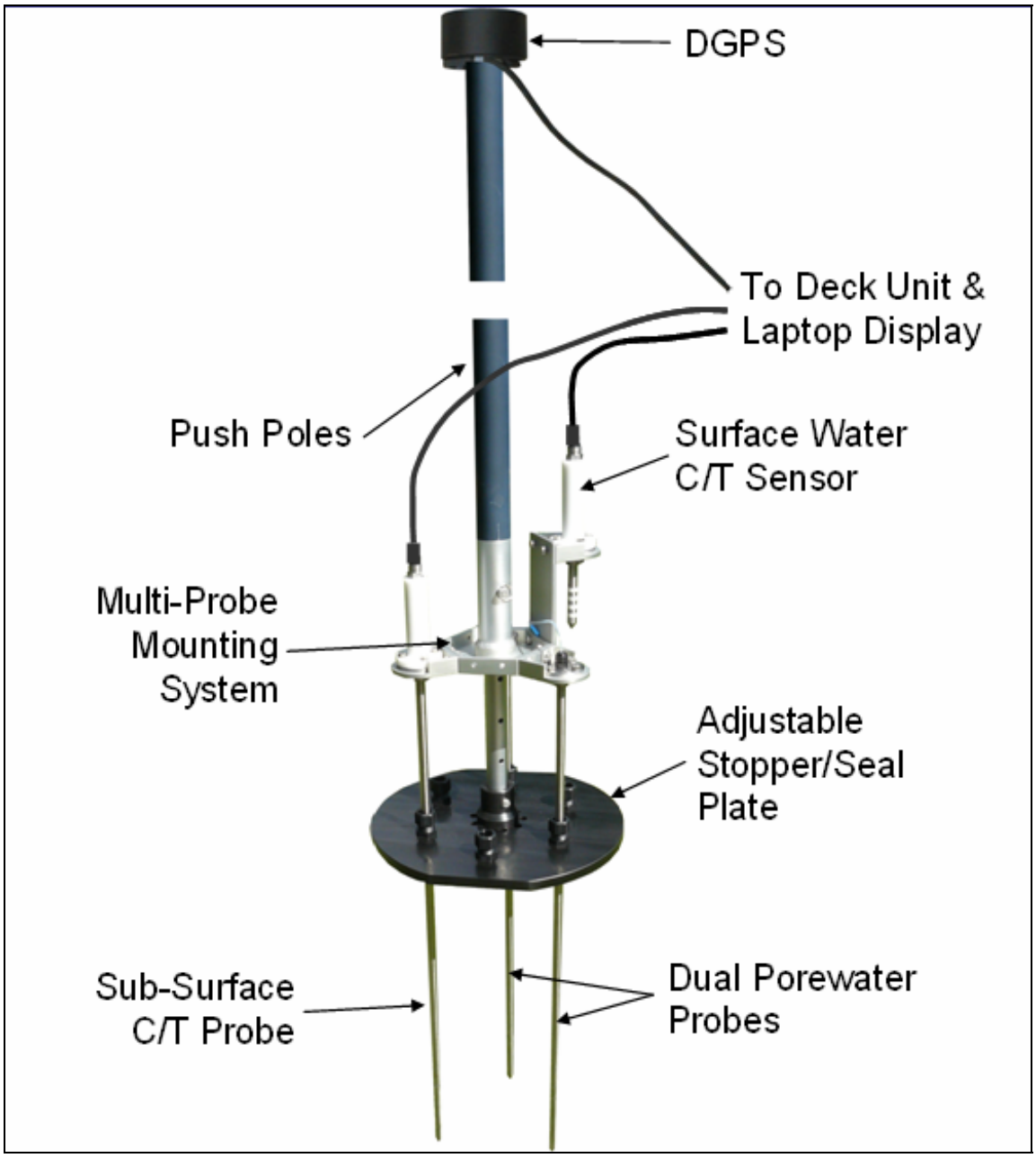

Figure 1. Complete Trident Probe showing sensor and water sampling probes, push-pole, DGPS unit.

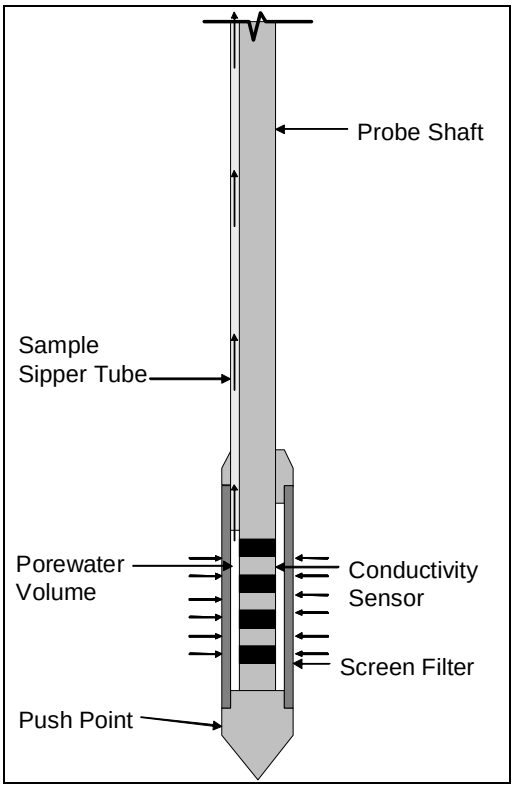

Figure 2. Schematic of the liquid-tip Trident conductivity probe.

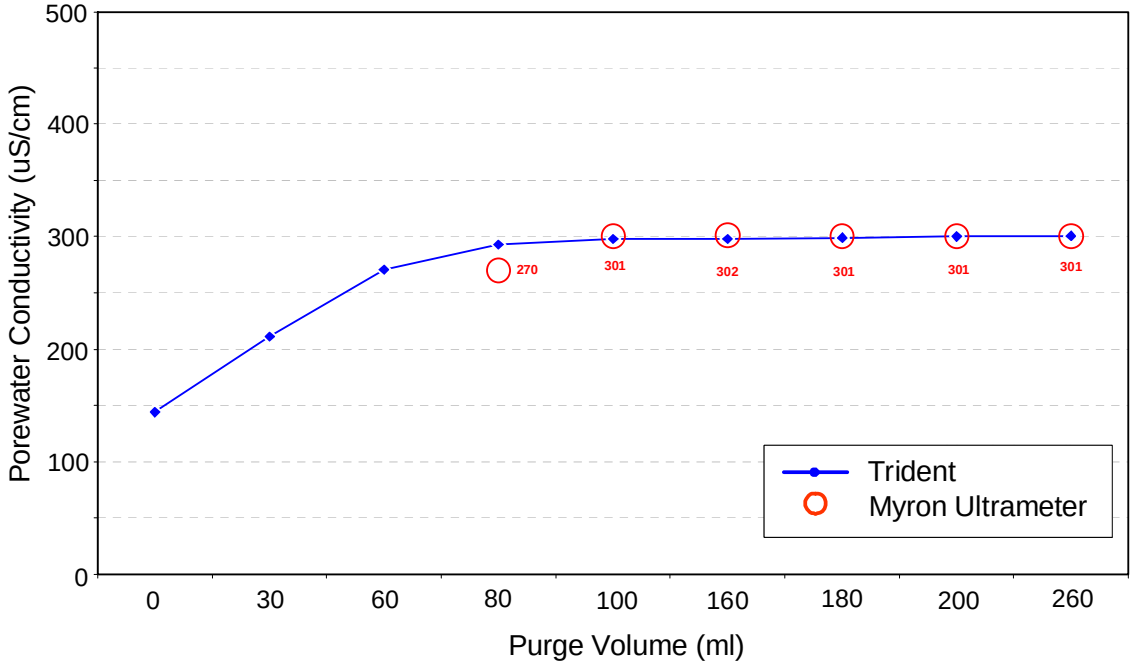

Figure 3. Typical porewater conductivity vs. purge volume for the liquid tip sensor.

Figure 4. Trident porewater system components.

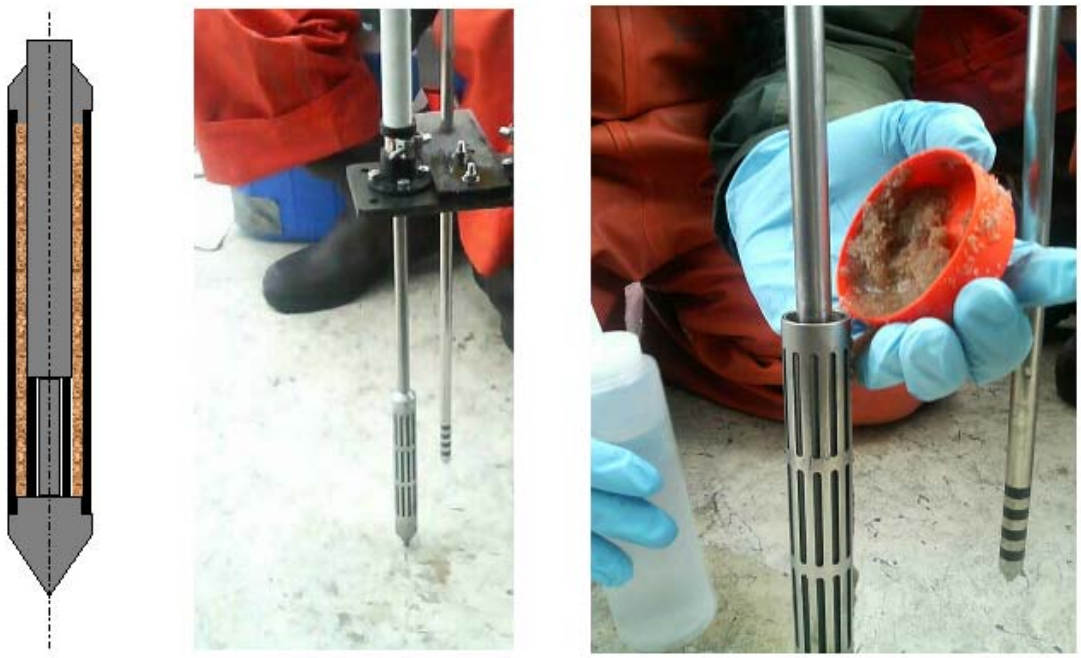

Figure 5. Trident porewater sampler with sand-pack system.

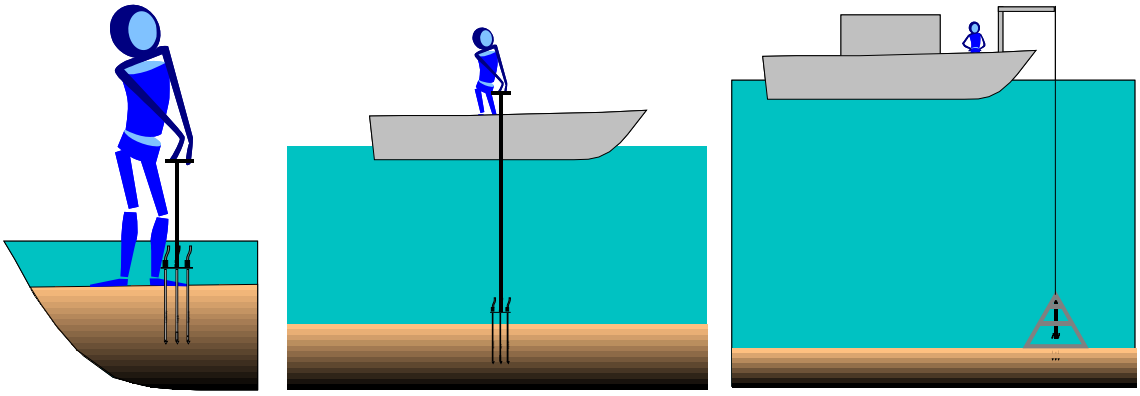

Figure 6. (Left to right) shallow-water (0 to 3 ft) push-pole, mid-range (3 to 30 ft) push-pole, and deep-water (>30 ft) deployment methods for Trident probe. Diver method not shown.

Figure 7. Typical shoreline survey grid for a Trident Probe survey.

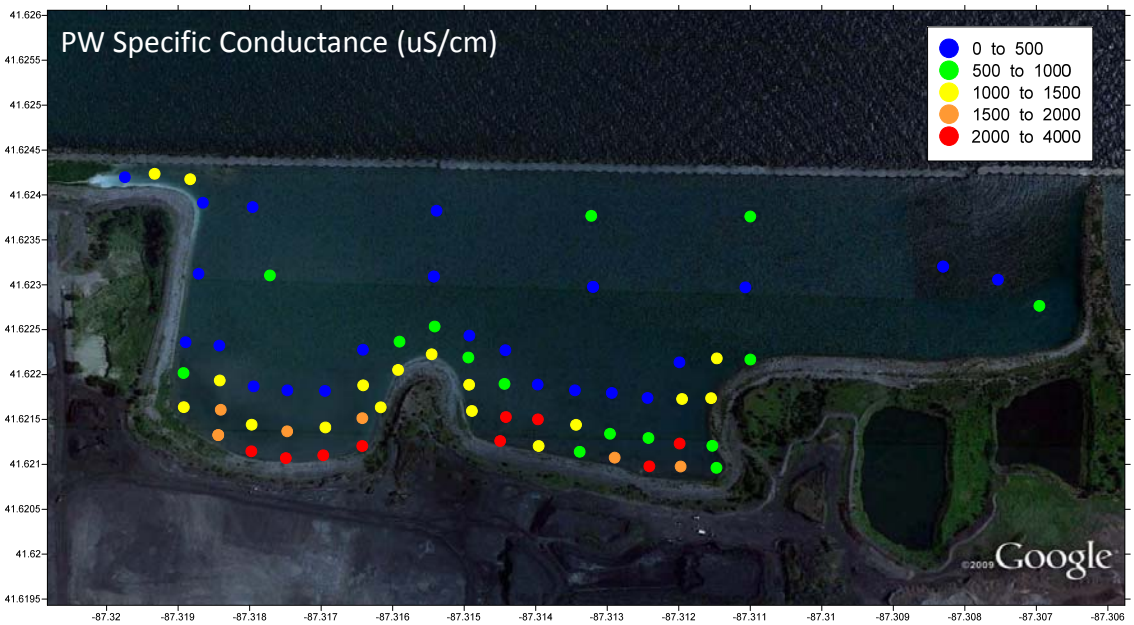

Figure 8. Example of liquid-tip sensor results for conductivity to detect groundwater discharge zones in a shoreline survey grid. In this case at a freshwater site, the groundwater was characterized by higher specific conductance levels than the surface water.

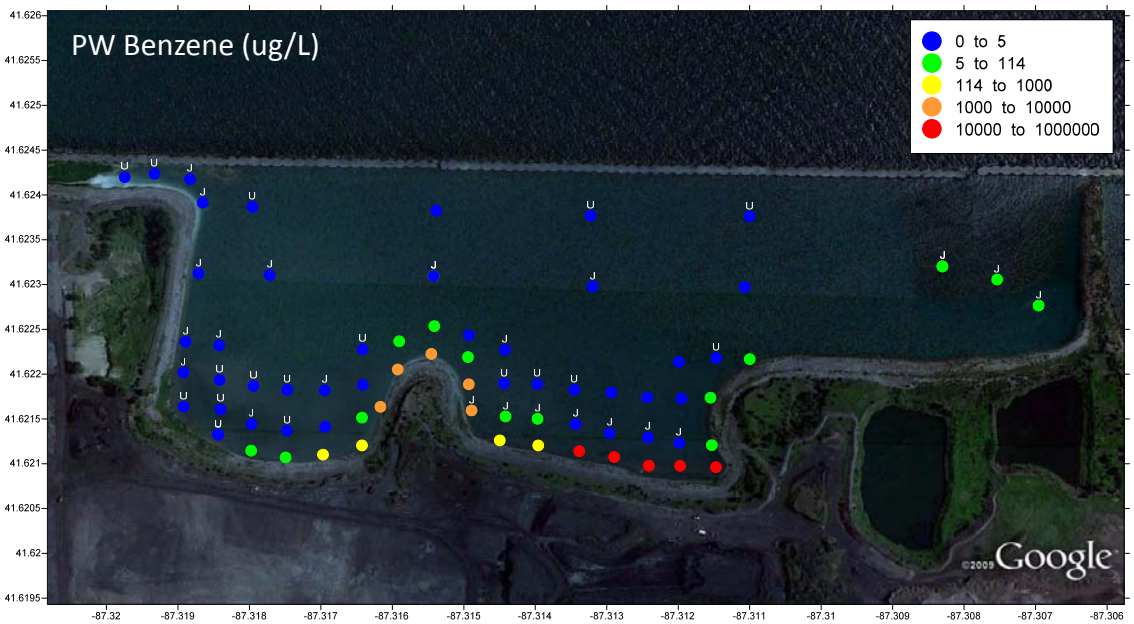

Figure 9. Example of porewater survey results for Benzene in a shoreline survey grid.